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A DIRECT PROCEDURE FOR DETERMINING THE NUMBER OF PLATES IN TACTOIDS OF SMECTITES: THE NA/CA-MONTMORILLONITE CASE*

I. SHOMER AND U. MINGELGRIN
Institute of Soils and Water, Agricultural Research Organization
The Volcani Center, POB 6, Bet Dagan, Israel

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Abstract—The dispersion of clays is of great importance in determining various soil properties such as hydraulic conductivity. A procedure which involves fixing followed by embedding of clay particles in an epoxy resin is described. This procedure enables the observation of cross sections of clay tactoids under a transmittance electron microscope, and the determination of the number of plates per tactoid. The use of the procedure for the determination of the relation between the exchangeable sodium percentage (ESP) and tactoid size in suspensions of a Na/Ca bentonite system is presented. It was demonstrated that even at ESP 5 significant dispersion already occurs, the average number of plates per tactoid being 6.6 as compared to 16.1 at ESP 0.

Key Words—Bentonite, Conductivity, Dispersion, Hydraulic, Smectite, Tactoid.

INTRODUCTION

The tactoid size of expandable clays is of great importance in determining the properties of soils in which such clays are present. Hydraulic conductivity, for example, is very sensitive to the tactoid size in aqueous suspension of the smectites present in the soil (Russo and Bresler, 1977). The adsorptive capacity of the expandable clays for various adsorbates (e.g., Albert and Harter, 1973) is another example of a property affected by the dispersion of the smectites, namely, their tactoid size.

In the past, a number of attempts have been made to establish the number of plates per tactoid in aqueous suspension. Viscosity (Shainberg and Otoh, 1968), light absorbance (Shainberg and Otoh, 1968; Banin and Lahav, 1968), and other data were used for indirect estimates of tactoid size. These attempts were only partially successful, as is evidenced, for example, by the discrepancies among the data reported by various investigators.

One important aspect of the problem is the dispersion ability of Na/Ca montmorillonites. When the exchangeable sodium percentage (ESP) reaches a certain critical point, the clay tends to disperse. From the viewpoint of soil improvement and management, the ESP to tactoid size relationship is of great interest. There are differences among the data in the various works reporting the tactoid size as a function of ESP (Shainberg and Otoh, 1968; Dufey et al., 1976; Shainberg and Kaiserman, 1969). This may be so because of the inaccuracies in the indirect estimates of the tactoid size used by these investigators, or because of some differences in the systems studied. At any rate, viscosity and light absorbance data were only qualitatively similar (Shainberg and Otoh, 1968).

In the present work a direct procedure to determine the tactoid size of smectites is reported. The ESP-tactoid size relation for a Na/Ca montmorillonite system is derived as an example of an important use for the reported procedure.

EXPERIMENTAL

Na- and Ca-homoionic montmorillonites were prepared from Fisher B-234 bentonite (Fisher Scientific Co., Fair Lawn, N.J., U.S.A.) by a procedure described previously (Shainberg and Otoh, 1968). After freeze-drying, 1% aqueous suspensions of the clay were shaken for 72 h. The suspensions consisted of mixtures of Na- and Ca-clays, with 0, 5, 10, 20, 40, and 100% Na-clay. Al-montmorillonite suspensions were also investigated. All aqueous suspensions were made with distilled water.

A drop of the clay suspension was mounted on a glass slide which was kept at 4°C, and immediately several drops of 2% aqueous solution of agar (Bacto-Agar, Difco Laboratories, Detroit, Michigan, U.S.A.) at 45°C were added around it. Immediate solidification of the agar followed. From the boundary region of the hardened agar drops in which the clay particles were now fixed, portions of approximately 2 mm^3 were transferred through ethanol-water solutions of increasing alcohol concentrations of 30, 50, 70, 90%. Agar portions were left for 15 min in each of the above solutions. Subsequently, these portions were kept for 1 h in analytical ethanol, with the ethanol being replaced every 15 min. The dehydrated agar portions were then embedded in low-viscosity epoxy resin (Spurr, 1969). Ultrasections were prepared with an ultratome (LKB-3). Observations and microphotographs were made by

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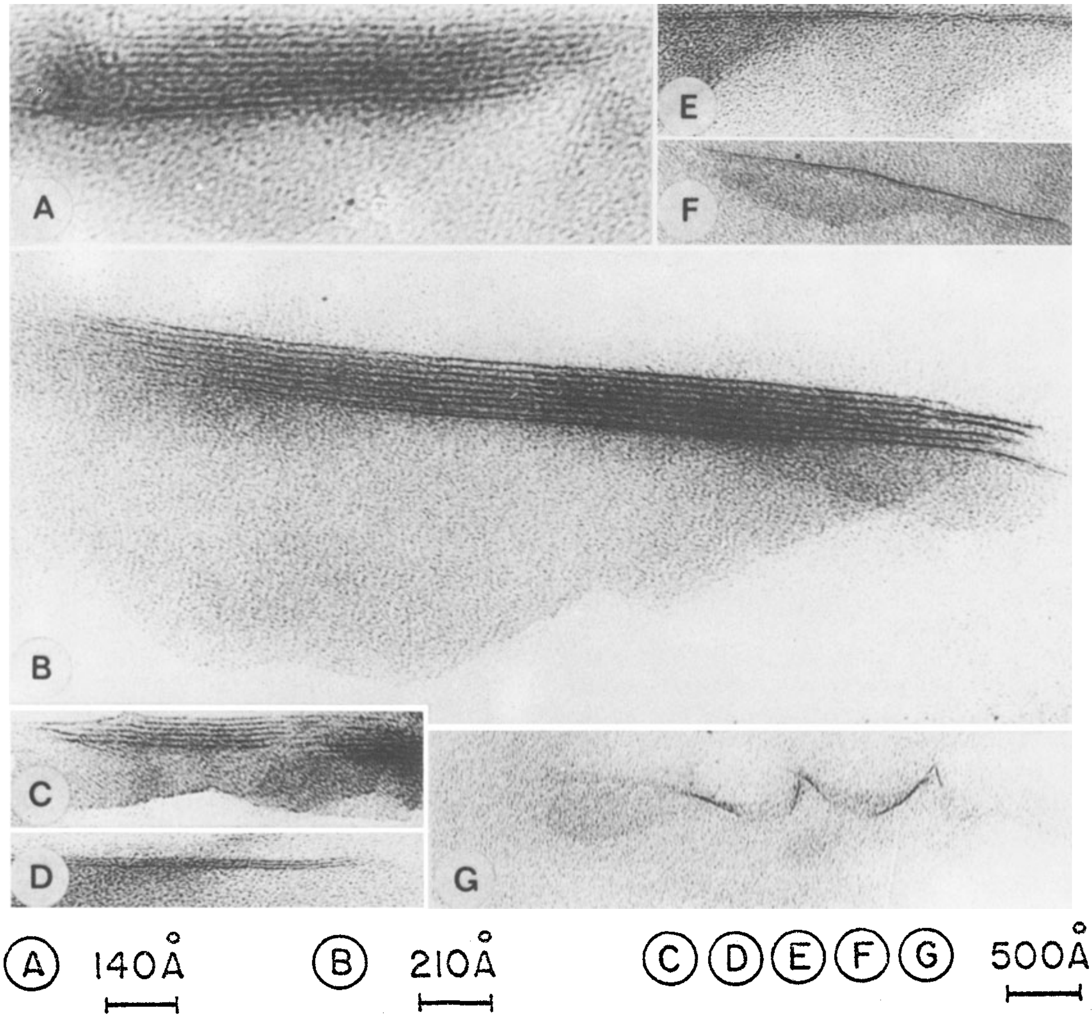

Fig. 1. Characteristic cross sections along the c-axis of Na/Ca bentonite tactoids. Figures 1A–G were taken from samples with ESP of 0, 5, 10, 20, 40, 100 and 100, respectively.

a Philips-300 transmission electron microscope (TEM). The magnifications used were 140,000 and above.

RESULTS AND DISCUSSION

The instantaneous hardening of the applied agar ensured that the clay particles embedded in it retained the tactoid size present in the aqueous suspension. The subsequent TEM observations indicated that the clay particles remained well defined. The procedure employed provided sections of all possible orientations and enabled the observation and actual counting of the plates.

Some characteristic cross sections along the clay's c-axis are presented in Figure 1A–F. The lamellar structure of the bentonites can be seen clearly. Another interesting feature is the wavy nature of many tactoids. Wavy tactoids were rather frequent; the thinner the tactoids, the greater their tendency to curve (Figure 1G). While in most cases curvature was not periodic, many tactoids were present with periodic undulations of wavelength of the order of magnitude of .01μm.

The size distribution of the tactoids is rather sharp, making the counting of the number of plates per tactoid statistically reliable. The average number of plates per tactoid is therefore also a meaningful parameter in the determination of various properties (e.g., hydraulic conductivity of smectite-containing soils). The only exception was the 5% Na system, in which the distribution was relatively wide. This may be a property of the Na/Ca system near the critical ESP where dispersion commences if such an ESP exists. Table 1 presents statistical data of the samples of tactoids counted.

In Figure 2 a plot of the average number of plates per tactoid vs. ESP is presented. It is clear that even 5% Na reduces sharply the number of plates per tactoid as compared with ESP 0. This is contrary to the findings

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Table 1. Statistical data concerning the size of tactoids in the Na/Ca montmorillonite at different ESP.

ESP	Average number of plates per tactoid	Standard deviation	Number of tactoids counted
0	16.1	1.5	14[1]
5	6.6	3.2	20
10	4.2	1.5	27
20	3.6	1.1	18
40	2.4	.9	15
100	1.4	.5	25

1. Aside from the tactoids counted, close to a hundred cross sections of tactoids were observed without counting. The distribution of the number of plates per tactoid about the average number reported here was narrow by inspection.

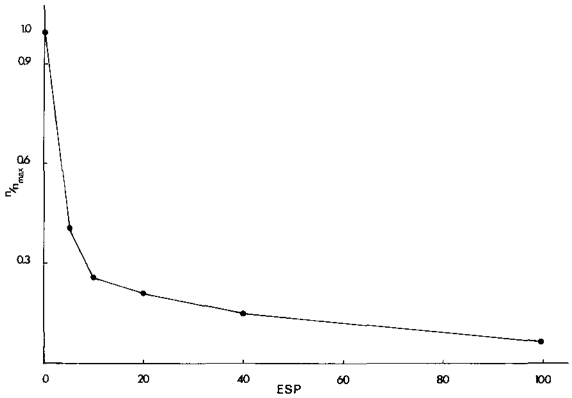

Fig. 2. The relative average number of plates per tactoid at various ESP. n is the number of plates per tactoid and n_{max} is the number of plates per tactoid at 100% Ca.

of Shainberg and Otoh (1968) and Shainberg and Kaiserman (1969), but in agreement with those of Dufey et al. (1976).

It is obvious that the electrolyte concentration in the aqueous clay suspensions as well as other factors will strongly affect the dispersion in a given ESP. The present results, therefore, should be taken only as an example of the use of the described procedure for determining the number of plates per tactoid.

In the past, TEM observations of the lamellar structure of smectites have been reported (e.g., Tchoubaric et al., 1973; Suito et al., 1969). The present procedure is outstanding in its ability to retain the tactoid size as present in the aqueous suspension, and in its ability to obtain easily a large number of cross sections along the c-axis. Thus, every system of interest (such as soils) can be analyzed by the present procedure to determine the dispersion of the smectites present in it.

One qualification should be made with thick tactoids such as those of the homoionic Ca-clay. Such tactoids may in some cases have two or even three outermost plates on each side shorter than the rest of the plates. The values given here are for centers of the cross sections, namely, the maximum number of plates.

The length of the tactoids, as expected from the mode of preparation, varied between .05 and 1μm, with most cross sections being around 0.2μm. It should be emphasized that since we observed cross sections, the maximum size of the tactoids might be somewhat larger.

Very few aggregates of a very thick cross section were observed in all preparations of about 50 plates per aggregate. These may be nondispersable aggregates due, for example, to some coating. Their large size makes these aggregates an important fraction of the bulk of the clay (up to 20%), despite their small number. However, for those properties where the dispersability of the clay is important, these aggregates are negligible. Such aggregates were not included in the distribution of tactoid size given in Figure 2 and Table 1.

Observations also were made on cross sections of Al-montmorillonite. Unlike the Na/Ca system, the Al-clay tended to appear in aggregates composed of a number of tactoids. Individual tactoids were composed of a large number of plates (around 25).

CONCLUSIONS

A simple and direct method was developed for determining the dispersion (or the number of plates per tactoid) of expandable clays. The procedure was employed successfully to determine the dispersion of a Na/Ca montmorillonite system. This procedure may be applied to any system containing expandable clay particles or other lamellar minerals.

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Резюме- Дисперсия глин имеет огромное значение при определении различных свойств почвы,таких как гидравлическая проводимость.Описывается процедура, которая включает фиксирование и внесение глинистых частиц в эпоксидную смолу.Эта процедура позволяет наблюдать профиль глинистых тактоидов под просвечивающим электронным микроскопом и определять число пластин на тектоид. Описывается испльзование процедуры для определения отношения между обменным количеством натрия /ОКН/ и размером тектоида в суспензиях бентонитовой системы Na/Ca.Было показано,что даже при ОКН=5 уже наблюдается значительная дисперсия,среднее число пластин на тактоид достигает 6,6 по сравнению с 16,1 при ОКН=0.

Kurzreferat- Die Dispersion von Tonerden ist von großer Wichtigkeit bei der Bestimmung verschiedener Eigenschaften wie z.B. hydraulische Leitfähigkeit. Eine Prozedur, welche Befestigung und nachfolgendes Einbetten von Ton partikeln in ein Epoxyharz umfasst, wird beschrieben. Dieses Verfahren macht die Observierung von Querschnitten von Tontaktoiden unter einem Trans parenzelektronenmikroskop und die Bestimmung der Anzahl von Tafeln per Taktoid möglich. Der Gebrauch dieser Prozedur für die Bestimmung des Verhältnisses zwischen dem austauschbaren-Natrium-Prozentsatz (ESP) und der Taktoid größe in Suspensionen eines Na/Ca Bentoniten wird presentiert.Es wurde demonstriert, daß sogar bei einem ESP- Wert von 5, schon Dispersion stattfindet. Die durchschnittliche Anzahl von Tafeln per Taktoid ist 6,6 verglichen mit 16,1 für einen ESP Wert von O.

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