

FACTORS AFFECTING THE MICROSTRUCTURE OF SMECTITES: ROLE OF CATION AND HISTORY OF APPLIED STRESSES

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Abstract—Small-angle X-ray scattering and transmission electron microscopy were used to determine the microstructure of Ca- and Na-smectite pastes. Samples were examined during drying and during rewetting at room temperature in an ultrafiltration cell by varying the suction pressure. The microstructure of Ca-smectite in a 10^{-3} M CaCl_2 at suction pressures corresponding to high water contents (≤ 1 bar) appears to consist of a network of quasi-crystals containing pores about $1 \mu\text{m}$ in size. The network walls result from the face-to-face bonding of 50 to about 400 layers, depending on the suction pressure and the previous degree of drying. For Na-smectites in a 10^{-3} M NaCl solution at low suction pressure (≤ 0.1 bar), the tactoids were found to consist of a few layers (≤ 10), and the water was found to be located essentially between the layers inside the tactoids.

Key Words—Microstructure, Small-angle X-ray scattering, Smectite, Suction pressure, Tactoid.

INTRODUCTION

As part of a study on the effects of wetting and drying of soils, Pons *et al.* (1982) previously reported on the structure of pastes of a Greek montmorillonite in both the sodium and the calcium forms containing different amounts of electrolyte solutions. Wetting and drying of the pastes was carried out at room temperature in a specially designed ultrafiltration cell (Tessier and Berrier, 1979). Structure determinations on the pastes was made using both transmission electron microscopy (TEM), and small-angle X-ray scattering (SAXS). The structure description comprised a description of texture (particle size and particle arrangement) as well as a description of the crystalline structure from the Ångström to the micrometer range to provide a better definition of particle and pore structure. In the present study, analogous experiments were carried out on various smectite pastes as a function of wetting and drying history in the ultrafiltration cell.

METHODS AND MATERIALS

The ultrafiltration cell in which isothermal drying and wetting cycles were carried out consists of a membrane supported by a metal grid at the bottom and an inlet for compressed air at the top, the pressure of which can be varied. The bottom of the cell was placed in a beaker containing water or an electrolyte solution. The clay paste was placed on the membrane so that the liquid in the beaker could be imbibed by the clay paste. Water take-up by the clay was governed by capillary action and by osmotic swelling. Together, these forces constitute the "suction" pressure of the system. The suction pressure at any stage of liquid uptake in the wetting and drying process is equal to the air pres-

sure applied above the paste. In the following suction pressure is expressed in bars. Curves of first drying of the pastes were obtained by applying air pressure ≤ 2.5 bar and then a relative vapor pressure to achieve equilibrium. Rehydration effects were studied by applying a maximum pressure to the clays as a drying step followed by rewetting at decreasing air pressures. At each equilibrium reached at a given pressure, the water content was measured by weighing the clay sample before and after heating to 150°C .

The following smectite clays were studied: Wyoming bentonite, a montmorillonite from Greece (layer charge 0.4 per half unit cell), and hectorite from Hector, California. From these clays in the sodium form, $<2\text{-}\mu\text{m}$ fractions were prepared by suspending the sodium clays in water, followed by repeated sedimentation. Both Na- and Ca-saturated suspensions were prepared in 10^{-3} M NaCl and 10^{-3} M CaCl_2 solutions, respectively, and homogenized by mechanical stirring.

Transmission electron microscopy (TEM) was carried out on ultrathin sections of about 500 Å thickness. These sections were prepared by successive replacements of the liquid phase by acetone, propylene oxide, and, finally an epoxy resin (Spurr, 1959; Tessier, 1984).

Small-angle X-ray scattering (SAXS) experiments were carried out with the X-rays emitted by the synchrotron storage ring at L.U.R.E.² The experimental conditions were described by Pons *et al.* (1981). Using such an X-ray source, the diagrams can be directly compared with theoretical SAXS curves. Computation models were used which assumed the particles to be

² L.U.R.E. = Laboratoire pour l'Utilisation du Rayonnement Electromagnétique.

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