



## Fractal analysis of surface roughness of montmorillonite clay self-supported films: Effects of exchanged cations and of mechanical tensile stress



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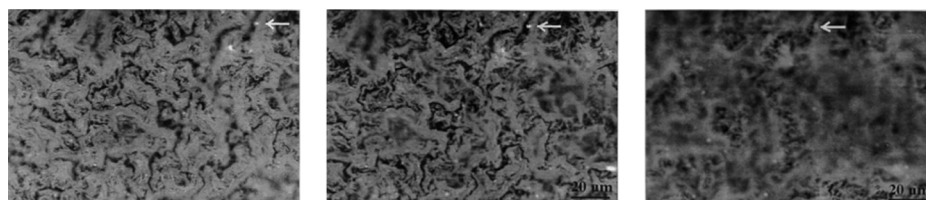
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### HIGHLIGHTS

- Charge and size of interfoliaire cation control films texture.
- The intensity of the surface forces controls the macroscopic mechanical properties.
- Mechanical loading shows pronounced plastic deformation for monovalent samples.
- Films with highly charged cations show stable roughness under mechanical load.
- Strong correlation of films surface H parameter with the parent cakes permeability.

### GRAPHICAL ABSTRACT



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### ABSTRACT

In this paper, surface roughness of self-supported montmorillonite clay films was studied by the use of fractal analysis to study its links with various inter-foliar cations, mechanical properties, and permeability. The Hurst exponent of fractional Brownian motion was estimated by maximum likelihood from optical images in reflection mode. Images corresponding to films with different inter-foliar exchanged cations visually show significant texture variations. It is confirmed by a continuous decrease of H parameter toward less correlated textures as one goes from the samples exchanged with monovalent ions to those exchanged with divalent and finally trivalent ions. The same films were analyzed while they were submitted to mechanical tensile stress. The films exchanged with the most charged cations or less hydrated ones do not show any evidence of texture change and the H parameter is almost constant. Concerning the permeability study, less permeable parent cakes – with hydrated monovalent cations – lead to relatively smooth and less disturbed surface films with high H parameter. In the opposite, very permeable cakes give birth to anti-persistent relief.

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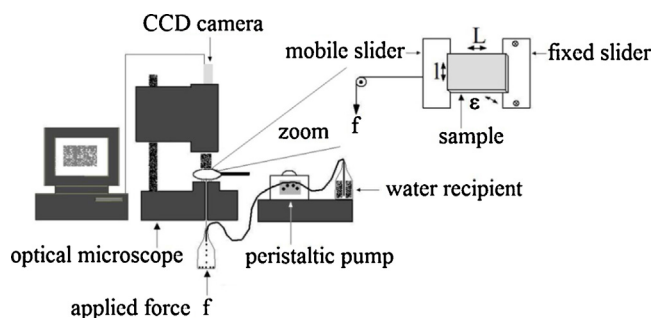
## 1. Introduction

The behavior of colloidal particles in suspensions is governed by two main factors: Brownian motion and surface forces. Removal

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**Fig. 1.** Homemade setup used for simultaneous mechanical and microstructural measurements.

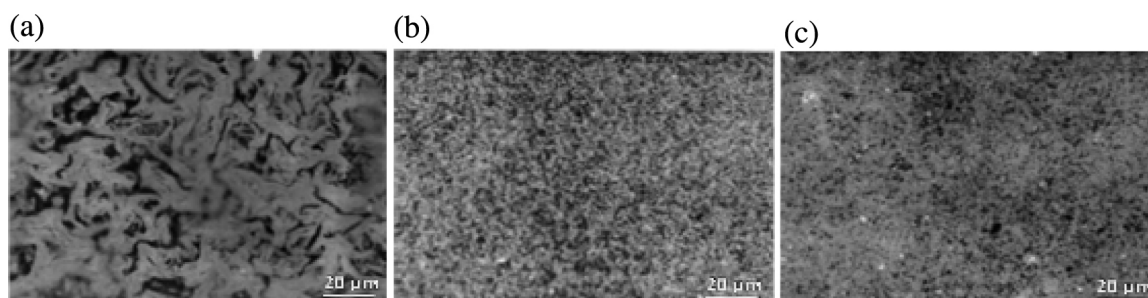
of the suspending medium leaves a solid material in which all Brownian motion has disappeared but in which the mechanical properties are still governed to a large extent by surface forces. Depending on the later and the drying procedure, the residual solid may be a fluffy powder, a highly porous and fragile monolithic gel, or a much more compact and cohesive solid. Examples of the later category are clays and hardened cement. The mechanical properties of colloidal solids like clays and cement have been extensively studied at macroscopic scale in terms of continuous stress-strain models in compaction or shear mode. However, little effort has been done to understand this behavior in terms of what might be called colloidal solid state physics, using concepts developed for atomic or molecular solids. One reason for this is the intrinsically disordered character of these materials, typical of granular media, which prevents the use of point or extended defect models such as dislocations for instance (although these may be used with highly ordered colloidal solids like latex particles crystals for instance). A second reason is the extreme difficulty of monitoring the individual particles motion during macroscopic strain. Many studies have been done to describe the microstructure of porous material by the use of fractal geometry. Pia et al. [1] have used an Intermingled Fractal Units model based on the Sierpinski carpet to simulate the experimental pore size distributions in porous material. A special analytical expression of the permeability has been established for a porous rock as a reference material. Some earth-based construction materials, natural or modified with additives also possess porous microstructure, the porosimetric properties can be geometrically described by means of models based on fractal geometry, where the fractal dimension of the experimental data set is determined following Pfeifer and Avnir's approach [2]. These models are useful for characterizing and understanding such materials. Modifications performed in order to improve water stability and compressive strength of this class of materials has effects on their surface erosion resistance [3]. However no correlation between the microstructure expressed as fractal dimension and such properties could be established. By

the use of Random packing simulations, Lee et al. [4] established a link between conventional structural parameters such as specific surface area, porosity, and non-conventional structural parameters such as fractal dimension, and permeability. Although these analyses are done on simple porous networks for establishing the correlations, this can be generalized to materials with high specific surface area and complex texture such as montmorillonite clay the material we studied in this paper. Synthesis of calcined alkaline earth phosphates (hydroxyapatites) synthesized via chemical precipitation, leads to porous materials with different textural characteristics. Through the nature of the earth metal, the surface hydroxyl group content is linked with surface fractal dimensions estimated by analyzing the N<sub>2</sub>-adsorption/desorption isotherms [5]. The biggest atom -Barium in this work- in hydroxyapatite shows irregular surface, indicating a high population of active sites across the surface. The same approach was followed in our work where a natural clay mineral is exchanged with different cations.

The present paper is an attempt to establish the link between the macroscopic mechanical behavior such as the inter-particle forces and the mesoscale reorganization of particles in a clay deposit under an applied stress through fractal analysis of the material surface. Rather than applying compressive and shear loading, as usual in soil and concrete mechanics, we choose to probe the tensile properties of thin colloidal solid samples, much in the same spirit as the studies performed on metallic materials, in which the mesoscale consequences of applying a tensile load on a sample are followed in situ in a transmission electron microscope. In our case, in view of the importance of maintaining the adsorbed water content of the clay samples for meaningful results, we used optical microscopy.

## 2. Smectite clays

Smectite clays [6,7] are polyelectrolytic inorganic colloids. Their primary particles are crystalline platelets with a thickness of 0.96 nm and a very large aspect ratio, which can go from about 25 for a synthetic smectite such as laponite, to more than 103 for some natural smectites such as montmorillonite, which will be used in the work. On the other hand, due to isomorphous ionic substitutions in the crystal lattice, the platelets bear a permanent negative charge of relatively low density, of the order of  $10^{-1} \text{ cm}^{-2}$ , compensated by hydrated and exchangeable cations. In the overwhelming majority cases, colloidal suspensions of large platelet smectites are not suspensions of individual platelets, in very dilute regime (volumic fraction less than  $10^{-3}$ ). In most cases, the particles in suspensions are ordered aggregates in which the platelets are parallel to each other and separated by a distance corresponding to a few water molecule diameters. Such aggregates were first termed quasi-crystals by Aylmore and Quirk [8]. The experimental evidence for ordered aggregates comes from a variety of measurements including X-ray diffraction [9], light scattering [10,11], viscosity measurements [11], swelling pressing measurements and calculations [12], anion exclusion [13], and NMR [14].



**Fig. 2.** Optical images of montmorillonite (Mmt) surface films exchanged with different cations. (a) Mmt-Li<sup>+</sup>, (b) Mmt-Ca<sup>2+</sup> and (c) Mmt-La<sup>3+</sup> Monovalent samples show relatively large texture elements which decrease in size when going towards trivalent ones.

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