

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

H. BEN RHAÏEM,¹ C. H. PONS,² AND D. TESSIER²

¹ Université d'Orléans, B.P. 4759, Rue de Chartres, 45067 Orléans Cédex 2

² Station de Science du Sol, I.N.R.A., 78000 Versailles, France

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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Proceedings of the International Clay Conference, 1985 Leonard Gene Schultz, H. Van Olphen, Frederick Albert Mumpton, 1987 Structural studies of clay minerals during 1981-1985 Extended x ray absorption fine structure study of cobalt exchanged sepiolite Infrared study of structural OH in kaolinite dickite and nacrite at 300 to 5 K Mixed layer minerals diffraction methods and structural features Hydration energies of smectites a model for glauconite illite and corrensite formation Formation of primitive clay precursors on K feldspar under extreme leaching conditions Origin of clay minerals in Pennsylvanian strata of the Illinois basin Clay minerals and sedimentary facies in the upper carboniferous pennine basin England a review Clay mineralogy of a weathered granophyre from north Queensland Australia Microtexture of clay rich sediments from the Oslofjord Norway Soil smectites and related interstratified minerals recent developments Classification of andisols in Japan based on physical properties Influence of noncrystalline material on phosphate adsorption by kaolin and bentonite clays Textural and mineralogical relationships between ferruginous nodules and surrounding clayey matrices in a laterite from Cameroon

Proceedings of the International Clay Conference Denver, 1985 LG Schultz (Ed), 1987

Proceedings of the International Clay Conference 1985, 1987 *Proceedings of the International Clay Conference 1985. Denver, Col., July 28 to August 2, 1985. Eds. Leonard G. Schultz, H. Van Olphen, Frederick A. Mumpton. Publ. by The Clay Minerals Society*, 1987

Handbook of Clay Science Faïza Bergaya, B.K.G. Theng, G. Lagaly, 2011-09-14 The first general texts on clay mineralogy and the practical applications of clay written by R E Grim were published some 40-50 years ago. Since then a vast literature has accumulated but this information is scattered and not always accessible. The Handbook of Clay Science aims at assembling the scattered literature on the varied and diverse aspects that make up the discipline of clay science. The topics covered range from the fundamental structures including textures and properties of clays and clay minerals through their environmental health and industrial applications to their analysis and characterization by modern instrumental techniques. Also included are the clay-microbe interaction, layered double hydroxides, zeolites, cement hydrates, genesis of clay minerals as well as the history and teaching of clay science. No modern book in the English language is available that is as comprehensive and wide-ranging in coverage as the Handbook of Clay Science. In providing a critical and up-to-date assessment of the accumulated information, this will serve as the first point of entry into the literature for both newcomers and graduate students, while for research scientists, university teachers, industrial chemists and environmental engineers, the book will become a standard reference text. Presents contributions from 66 authors from 18 different countries who have come together to produce the most comprehensive modern handbook on clay science. Provides up-to-date concepts, properties and reactivity of clays and clay minerals in a one-stop source of information. Covers classical and new environmental, industrial and health applications of clays as well as the instrumental techniques for clay mineral analysis. Combines geology, mineralogy, crystallography with physics, geotechnology and soil mechanics together with inorganic

organic physical and colloid chemistry for a truly multidisciplinary approach

Clay Mineral Catalysis of Organic Reactions Benny K.G Theng, 2018-07-27 The book provides insight into the working of clays and clay minerals in speeding up a variety of organic reactions Clay minerals are known to have a large propensity for taking up organic molecules and can catalyse numerous organic reactions due to fine particle size extensive surface area layer structure and peculiar charge characteristics They can be used as heterogeneous catalysts and catalyst carriers of organic reactions because they are non corrosive easy to separate from the reaction mixture and reusable Clays and clay minerals have an advantage over other solid acids as they are abundant inexpensive and non polluting

Handbook of Clay Science, 2013-07-23 The first edition of the Handbook of Clay Science published in 2006 assembled the scattered literature on the varied and diverse aspects that make up the discipline of clay science The topics covered range from the fundamental structures including textures and properties of clays and clay minerals through their environmental health and industrial applications to their analysis and characterization by modern instrumental techniques Also included are the clay microbe interaction layered double hydroxides zeolites cement hydrates and genesis of clay minerals as well as the history and teaching of clay science The 2e adds new information from the intervening 6 years and adds some important subjects to make this the most comprehensive and wide ranging coverage of clay science in one source in the English language Provides up to date comprehensive information in a single source Covers applications of clays as well as the instrumental analytical techniques Provides a truly multidisciplinary approach to clay science

Paleozoic sequence stratigraphy; views from the North American Craton Brian J. Witzke, Greg A. Ludvigson, Jed Day, 1996-01-01

Microstructure of Fine-Grained Sediments W.A. Chiou, Richard H. Bennett, R.W. Faas, William R. Bryant, J. Kasprovicz, Matthew H. Hulbert, H. Li, T. Lomenick, N.R. O'Brien, S. Pamukcu, P. Smart, C.E. Weaver, T. Yamamoto, 2012-12-06 Knowledge of basic clay microstructure is fundamental to an understanding of the physical chemical and mechanical properties of fine grained sediments and rocks This compilation of fifty nine peer reviewed papers examines clay microstructure in detail with comprehensive sections focusing on microstructure signatures environmental processes modeling measurement techniques and future research recommendations Many of these topics are discussed in light of geological and engineering applications such as hazardous waste disposal construction techniques and drilling programs The field of clay microstructure is developing rapidly The concepts observations and principles presented in this book will help stimulate new thought and be a spring board for exciting new research

Clays: Controlling the Environment GJ Churchman, RW Fitzpatrick, RA Eggleton, 1995-01-01 Proceedings of the 10th International Clays Conference Adelaide Australia July 18 to 23 1993 Clays have provided us with the most active ingredients in soils with building materials with pottery and ceramics for both utility and decoration and with coatings and fillers for paper among other uses The unique properties of these apparently everyday materials are being studied and used in an increasing range of industrial and environmental applications Clays Controlling the Environment provides a valuable compendium of the latest

results from the complete range of clay related scientific research It includes coverage of the economic and environmental issues as well as directions for further research and development in many vital and expanding industries All papers in these proceedings were subject to peer review The topics discussed are Clays in industry and the environment Surface and interlayer reactions Clay mineral structures and chemistry Methods of investigation Clays in geology Soil mineralogy The emphasis of this book reflects the vital role that clays play in controlling natural polluted and technological environments

Chemical Physics of Intercalation II Patrick Bernier, John E. Fischer, Siegmund Roth, Stuart A. Solin, 2012-12-06 This volume provides a record of the second ASI on the subject Chemical Physics of Intercalation which was patterned after its highly successful July 1987 predecessor A growing community of chemists physicists and materials scientists has come to appreciate the utility of extending the intercalation concept to generic guest host compounds and solid solutions The unifying themes are the complex phase equilibria which result from the competition between repulsive and attractive interactions between and within the guest and host substructures the tunability of properties by control of guest concentration and superlattice periodicity and the broad spectrum of potential applications which these materials may provide The success of this initiative may be judged by noting the enlarged scope of materials covered in this volume as compared to its predecessor The present volume covers the spectrum from 3 dimensional oxides 2 dimensional classical layer intercalates dimensional doped polymers and zero dimensional doped fullerene lattices Hybrid systems such as polymers in layer hosts and nonporous hosts are also treated Several chapters provide global unifying viewpoints by focussing on solid state chemical aspects transport and optical properties the occurrence of superconductivity etc

Index of Conference Proceedings Received British Library. Document Supply Centre, 1987 **Manual of Physico-Chemical Analysis of Aquatic Sediments** Alena Mudroch, Jose M. Azcue, Paul Mudroch, 2017-10-05 Because water is one of the most important life supporting media on the planet the quality of aquatic ecosystems is of great interest to the entire world population One of the factors that greatly affects water quality is the condition of the underlying sediment layer The Manual of Physico Chemical Analysis of Aquatic Sediments addresses the best methods for quantitative determination of chemical forms of different elements and compounds bioassessment techniques and determination of physical properties of sediments Essential information for surveying research and monitoring of sediment contamination is covered This manual will aid sediment biologists geochemists limnologists regulatory program managers environmental chemists and toxicologists and environmental consultants in preparing plans for proper remedial action

Soil Clays G. Jock Churchman, Bruce Velde, 2019-06-10 As the human population grows from seven billion toward an inevitable nine or 10 billion the demands on the limited supply of soils will grow and intensify Soils are essential for the sustenance of almost all plants and animals including humans but soils are virtually infinitely variable Clays are the most reactive and interactive inorganic compounds in soils Clays in soils often differ from pure clay minerals of geological origin They provide a template for most of the reactive organic matter in soils They directly affect plant nutrients

soil temperature and pH aggregate sizes and strength porosity and water holding capacities This book aims to help improve predictions of important properties of soils through a modern understanding of their highly reactive clay minerals as they are formed and occur in soils worldwide It examines how clays occur in soils and the role of soil clays in disparate applications including plant nutrition soil structure and water holding capacity soil quality soil shrinkage and swelling carbon sequestration pollution control and remediation medicine forensic investigation and deciphering human and environmental histories Features Provides information on the conditions that lead to the formation of clay minerals in soils Distinguishes soil clays and types of clay minerals Describes clay mineral structures and their origins Describes occurrences and associations of clays in soil Details roles of clays in applications of soils Heavily illustrated with photos diagrams and electron micrographs Includes user friendly description of a new method of identification To know soil clays is to enable their use toward achieving improvements in the management of soils for enhancing their performance in one or more of their three main functions of enabling plant growth regulating water flow to plants and buffering environmental changes This book provides an easily read and extensively illustrated description of the nature formation identification occurrence and associations measurement reactivities and applications of clays in soils *Developments in Palygorskite-Sepiolite Research* ,2011-07-20 The aim of this comprehensive book is to present the most important results achieved in the research of the clay minerals palygorskite and sepiolite Palygorskite and sepiolite have found to be useful in a huge variety of industrial and medical applications As a result research on these clays has been intensified during the last two decades and important advances in their characterization have been made The book contains contributions from distinguished scientists in the field Comprehensive treatment of palygorskite and sepiolite Cutting edge developments in industrial minerals and applications Written by distinguished scientists in the field Organo-Clay Complexes and Interactions Shmuel Yariv,Harold Cross,2001-11-02 Provides comprehensive coverage of the structures properties and interactions of organo clay complexes as well as their role in the origin of life Presents current techniques in nuclear magnetic resonance differential thermal analysis and thermogravimetry visible spectroscopy and infrared and thermal infrared spectroscopy for the analysis of fine structures in organo clay complexes **Hybrid Organic-Inorganic Interfaces** Marie Helene Delville,Andreas Taubert,2017-12-04 Hybrid organic inorganic materials and the rational design of their interfaces open up the access to a wide spectrum of functionalities not achievable with traditional concepts of materials science This innovative class of materials has a major impact in many application domains such as optics electronics mechanics energy storage and conversion protective coatings catalysis sensing and nanomedicine The properties of these materials do not only depend on the chemical structure and the mutual interaction between their nano scale building blocks but are also strongly influenced by the interfaces they share This handbook focuses on the most recent investigations concerning the design control and dynamics of hybrid organic inorganic interfaces covering i characterization methods of interfaces ii innovative computational approaches and simulation of

interaction processes iii in situ studies of dynamic aspects controlling the formation of these interfaces and iv the role of the interface for process optimization devices and applications in such areas as optics electronics energy and medicine

Quantitative Diagenesis: Recent Developments and Applications to Reservoir Geology A. Parker,B.W.

Sellwood,2012-12-06 Reservoirs generally consist of sandstones or carbonates exhibiting heterogeneities caused by a wide range of factors Some of these formed depositionally e g as channels palaeosols clay seams or salts others may be diagenetic in origin e g carbonate or silica cemented zones authigenic clays karstic surfaces The severity with which diagenesis affects rock systems results from the interplay between the diagenetic process itself and the timescale over which it operated The book provides a wide ranging overview of diagenetic processes and responses in calcareous argillaceous arenaceous and carbon rich microbial and organic sedimentary systems It introduces diagenetic concepts reviews existing knowledge and shows how existing qualitative approaches might be developed in more quantitative ways Several chapters consider mass balance calculations and the temporal and spatial aspects of diagenetic processes It is unique as a textbook in providing such a breadth of diagenetic subject range and such depth of coverage in each topic It provides a source reference for advanced students and professionals active in reservoir and aquifer studies

Development of Pedotransfer Functions in Soil

Hydrology Yakov Pachepsky,W.J. Rawls,2004-12-30 Environmental and agricultural modeling and assessment have a multitude of uses for soil parameters governing retention and transport of water and chemicals in soils These parameters are notorious for the difficulties and high labor costs involved in measuring them Good estimates instead of direct measurements may be accurate enough for many applications Pedotransfer functions provide such estimates by utilizing available soil survey information to translate data we have into data we need This book is the first book on the topic It provides the unique compendium of pedotransfer functions summarizes the vast international experience in this field and shows how the value of soil data can be increased by using them in pedotransfer functions to predict soil hydrologic and related properties The book is a rich source of information crucial for environmental research and applications

Geochemistry of Clay-Pore Fluid

Interactions D.A.C. Manning,P.L. Hall,C.R. Hughes,1993-02-28 30% discount for members of The Mineralogical Society of Britain and Ireland This book contains review papers covering such issues as the geometrical characterization of porous solids and particle aggregates using synchrotron radiation techniques A review of the phenomenon of overpressuring primal global distribution of clay minerals and the use of clays as barriers in waste disposal are also included

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