

FEBEX
FULL-SCALE ENGINEERED BARRIERS EXPERIMENT
IN CRYSTALLINE HOST ROCK

BENTONITE:
ORIGIN, PROPERTIES
AND FABRICATION OF BLOCKS

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DE LOS RECURSOS NATURALES (AITEMIN)*

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LIST OF SOME ABBREVIATIONS/ACRONYMS USED

AITEMIN	Asociación para la Investigación y Desarrollo Industrial de los Recursos Naturales (Spanish association for industrial research and development of natural resources)
CD	Consolidated Drained triaxial test
CEA	Commissariat à l'Énergie Atomique (French atomic energy commission)
CEC	Cation Exchange Capacity
CIEMAT	Centro de Investigaciones Energéticas, Medio Ambientales y Tecnológicas (Spanish center of technology, environmental and energy research)
CSIC-Barcelona	Consejo Superior de Investigaciones Científicas - Instituto Jaume Almera (Spanish national agency of scientific research - Jaume Almera Institute at Barcelona)
CSIC-Zaidín	Consejo Superior de Investigaciones Científicas - Estación experimental del Zaidín (Spanish national agency of scientific research - experimental station at Zaidín)
CU	Consolidated Undrained triaxial test
DTA	Differential Thermal Analysis
E.C.	Electric Conductivity
EDTA	Ethylene Diamine Tetra acetic Acid (sodium salt)
ENRESA	Empresa Nacional de Residuos Radiactivos (Spanish national company for radioactive waste)
EU	European Union
FEBEX	Full-Scale Engineered Barrier Experiment in Crystalline Host Rock
HLW	High-Level radioactive Waste
MPN	Most Probable Number
NAGRA	Nationale Genossenschaft für die Lagerung Radioaktiver Abfälle (Swiss national cooperative for the storage of radioactive wastes)
OA	Oriented clay mineral Aggregate
OA+EG	Oriented clay mineral Aggregate treated with Ethylene Glycol
R&D	Research and Development
TGA	Thermogravimetric Analysis
THG	Thermo-Hydro-Geochemical
THM	Thermo-Hydro-Mechanical
UAM	Universidad Autónoma de Madrid (Spanish Autonomous University of Madrid)
UPC-DIT	Universidad Politécnica de Cataluña - Departamento de Ingeniería del Terreno (Spanish Polytechnical University of Cataluña - Ground Engineering Department)
XRD	X-Ray Diffraction

RESUMEN

El Proyecto FEBEX (Full-scale Engineered Barriers Experiment) tiene como propósito el estudio del comportamiento de componentes del campo próximo de un almacenamiento de residuos radioactivos de alta actividad en roca cristalina, según un concepto en el que la barrera de ingeniería entre el contenedor de residuos y la roca almacén está constituida por bloques de bentonita compactada. El FEBEX consta de un ensayo "in situ", en condiciones naturales y escala real (Grimsel, Suiza); un ensayo en "maqueta" (CIEMAT, Madrid), a escala casi real; y una serie de ensayos de laboratorio para complementar la información de los dos ensayos a gran escala. Durante la etapa preoperacional del Proyecto se ha realizado la planificación, diseño e instalación de los ensayos a gran escala, la modelación predictiva de su comportamiento y la caracterización básica de la bentonita.

Este informe es uno de los correspondientes a la fase preoperacional del Proyecto FEBEX, y se refiere a la selección y caracterización de la bentonita FEBEX, así como a la fabricación de los bloques que se han utilizado en los ensayos "in situ" y en "maqueta".

La arcilla utilizada proviene del yacimiento Cortijo de Archidona (Almería), de donde también se extrajo la arcilla S-2, utilizada en otros proyectos de I+D de ENRESA anteriores al FEBEX. Los resultados obtenidos en estos proyectos se resumen en el capítulo 2, ya que algunos parámetros utilizados en la modelización predictiva se han tomado de los de la arcilla S-2, por no haberse determinado todavía para la arcilla FEBEX.

Para el Proyecto FEBEX se han seleccionado, homogeneizado y procesado unas 300 toneladas de bentonita, cuyo tratamiento en fábrica ha consistido en disgregación mecánica y secado suave hasta humedades alrededor del 14 %, rechazándose todo el material de tamaño superior a 5 mm. Este material se ha utilizado para la fabricación de los bloques de los dos ensayos a gran escala y en los ensayos de laboratorio. Se han tomado varias muestras de cada lote de procesado, por mezcla de las cuales se ha obtenido una muestra única, de referencia 70-IMA-3-4-0, con la que se ha trabajado en los laboratorios participantes. La caracterización mineralógica y química se ha llevado a cabo en CIEMAT y CSIC-Zaidín, y la caracterización mecánica, hidráulica y térmica se ha realizado en CIEMAT y UPC-DIT, habiéndose obtenido resultados concordantes entre todos los laboratorios.

La bentonita FEBEX tiene un contenido en montmorillonita superior al 90 %, con cuarzo, plagioclasa, feldespato potásico, calcita y tridimita como minerales accesorios. Es posible que existan interstratificados illita/esmectita en proporciones de hasta el 15 %. La capacidad de cambio catiónico es superior a 100 meq/100g, y en el complejo de cambio aparece 42 % de Ca, 33 % de Mg, 23 % de Na y 2 % de K.

Las propiedades de identificación no corresponden a las esperadas para un material de esta mineralogía, ya que tanto el límite líquido (100 %) como la superficie específica externa (32 m²/g) son algo bajos y la proporción de fracción menor de 2 µm obtenida después de una dispersión intensa es sólo del 65 %, lo que hace suponer que se produce algún tipo de aglutinación de las partículas (quizá por cemento silíceo), cuyas consecuencias sobre el comportamiento de la arcilla no han sido evaluadas.

Se ha determinado la presión de hinchamiento y la conductividad hidráulica saturada de pastillas compactadas a densidad seca de 1,60 y 1,70 g/cm³, así como la conductividad térmica para estas densidades y diferentes grados de saturación. Se ha determinado también la relación succión/humedad y una superficie de estado preliminar para succiones decrecientes y con cambios pequeños de tensiones.

Los resultados obtenidos muestran ligeras variaciones respecto a los valores obtenidos para la arcilla S-2, con una tendencia de la arcilla FEBEX a ser más impermeable y desarrollar mayor presión de hinchamiento. Sin embargo, de cara a la evaluación del comportamiento de un almacenamiento (PA), se considera que estas diferencias entran dentro del intervalo de variación esperado para las propiedades de la bentonita.

Se han fabricado dos conjuntos de bloques: uno para el ensayo en "maqueta", bajo la dirección y control de CIEMAT, y otro para el ensayo "in situ", bajo la dirección y control de AITEMIN. En ambos casos se ha recurrido a un diseño en coronas concéntricas que redujera al mínimo el volumen de huecos de la barrera. Los bloques se han fabricado mediante compactación uniaxial a presiones de 40 a 50 MPa, obteniéndose una densidad seca media de los bloques para el ensayo en "maqueta" en torno a 1,77 g/cm³ y de los bloques para el ensayo "in situ" en torno a 1,70 g/cm³. Esto da lugar a una densidad seca media de la barrera instalada de 1,65 y 1,60 g/cm³, respectivamente.

EXECUTIVE SUMMARY

The aim of the FEBEX (Full-scale Engineered Barriers Experiment) Project is to study the behavior of the near field components of a high-level radioactive waste (HLW) repository in crystalline rock, in accordance with a concept in which the canisters are surrounded by a clay barrier constructed of highly-compacted bentonite blocks. The FEBEX consists of a full scale "in situ" test (Grimsel, Switzerland) performed under natural conditions; a "mock-up" test (CIEMAT, Madrid), at almost full scale; and a series of laboratory tests designed to complement the information from the two large-scale tests.

During the pre-operational stage of the Project, the planning, design and installation of the two large-scale tests was accomplished, as well as the predictive modelling of their behavior and the basic characterisation of the bentonite. This report is one of those concerning the pre-operational stage of the Project, and refers to the selection and characterisation of the FEBEX bentonite, and to the fabrication of the blocks used in the "in situ" and "mock-up" tests.

The clay utilized comes from the Cortijo de Archidona deposit (Almería), from where the S-2 clay used in previous ENRESA R&D Projects was also extracted. The results obtained in these Projects are summarised in Chapter 2, as some of the parameters considered in the predictive modelling were taken from the S-2 bentonite database, due to their not having yet been determined for the FEBEX clay.

For the FEBEX Project, about 300 tons of bentonite have been selected, homogenized and treated. At the factory, the clay was disaggregated and gently dried to a water content of around 14 %, all the material of particle size greater than 5 mm being rejected. The processed material was used for fabrication of the blocks for the two large-scale tests and for the laboratory tests. Samples of each processing lot were taken, and were mixed to obtain a single sample, whose reference is 70-IMA-3-4-0, which is the one used by all the participant laboratories. Mineralogical and chemical characterisation was performed at CIEMAT and CSIC-Zaidín, and the mechanical, hydraulic and thermal characterisation was carried out by CIEMAT and

UPC-DIT; the results obtained at each laboratory were consistent with the rest of the laboratories.

The FEBEX bentonite has a montmorillonite content greater than 90 %, with quartz, plagioclase, potassic feldspar, calcite and trydimite as accessory minerals. There may be illite/smectite interstratifications up to 15 %. The cation exchange capacity is higher than 100 meq/100g, and in the exchange complex 42 % Ca, 33 % Mg, 23 % Na and 2 % K are present.

The identification properties are not exactly those expected for a material with this mineralogy. Both the liquid limit (100 %) and the external specific surface (32 m²/g) are rather low, and the less than 2 µm fraction obtained after intense dispersion amounts to only 65 %. This might mean that the particles are agglutinated in a certain way (possibly by a siliceous cement), although the consequences of this on the behavior of the clay have not been evaluated.

Swelling pressure and saturated hydraulic conductivity were obtained for specimens compacted to dry densities of 1.60 y 1.70 g/cm³, along with the thermal conductivity for these densities and different degrees of saturation. The suction/water content relationship and a preliminary state surface for decreasing suctions and small stress changes were determined.

The results obtained show slight variations with respect to the values obtained for the S-2 clay, with a tendency of the FEBEX clay to be more impervious and to develop a higher swelling pressure. However, for the performance assessment of a repository (PA), these differences are considered to be in the expected range of variation of the bentonite properties.

Two sets of blocks have been manufactured: one for the "mock-up" test, under the management and supervision of CIEMAT, and another for the "in situ" tests, under the management and supervision of AITEMIN. In both cases a crown design has been adopted, in order to minimize the void volume of the barrier. The blocks have been uniaxially compacted to pressures of 40 to 50 MPa, the resulting dry density being 1.77 g/cm³ for the blocks for the "mock-up" test and 1.70 g/cm³ for the blocks for the "in situ" test. Consequently, the average dry density of the installed barrier is 1.65 and 1.60 g/cm³, respectively.

1

INTRODUCTION

The aim of FEBEX (Full-scale Engineered Barriers Experiment) is the study of the behavior of components of the near field for a repository of high-level radioactive wastes (HLW) in crystalline rock. The experiment consists of three principal parts: 1) an "in situ" test, in natural conditions and at full scale [1]; 2) a "mock-up" test, at almost full scale [2]; and 3) a series of laboratory tests to complement the information from the two large-scale tests [3, 4]. The experiment is based on the Spanish disposal reference concept for crystalline rock (AGP Granito): the waste canisters are placed horizontally in drifts and surrounded by a clay barrier constructed from highly-compacted bentonite blocks [5].

The project will last about 7 years (1994 through 2001) and its execution has been divided into four successive stages, defined by the principal tasks to be performed in the two large-scale tests. These stages are: pre-operational (planning, design, characterization of the clay, installation, and modeling); operational (heating, monitoring, cooling, and verification of predictions); dismantling (extraction, inspection, sampling, and study of the materials); and final evaluation of the results and verification of the models.

This report is a summary of the work performed in the pre-operational stage (from the middle of 1994 to the beginning of 1997).

The engineered barriers (waste, canister, and clay barrier) are key elements in the final disposal concept for the HLW. The matrix of the waste acts as a reducing barrier that minimizes the solubility; the canister isolates the waste, during a certain time, and contributes to the reducing environment. The clay barrier has the multiple purpose of providing mechanical stability to the canister, by absorbing stress and deformations; of sealing the discontinuities of the adjacent rock and retarding the arrival of the groundwater at the canister; and of retaining/retarding the migration of the radionuclides released, once the failure of the canister and the oxidation/dissolution of the combustible matrix are produced.

The behavior of a HLW repository is determined, in great measure, by the characteristics of the design and construction of the engineered barriers and especially by the changes that may be produced in the mechanical, hydraulic, and geochemical properties as a result of the combined effects of heat generated by the radioactive decay and of the water and solutes contributed from the surrounding rock. Therefore, it has been consid-

ered, that it is fundamental to comprehend and quantify the processes that take place in the near field to evaluate its long-term behavior.

ENRESA has studied, in prior R&D plans, sources of supply of materials to be used in the clay barrier, as well as their thermal, hydraulic, mechanical, and geochemical behavior. Integral characterization studies of granite masses have also been made. As a consequence of these studies, and the fact that, until now, there has not been made any large-scale experiment in granite with the horizontal disposition of the waste canisters in drifts, ENRESA has reached the conclusion that the next step, in establishing the viability of the concept and in progressing in the understanding and evaluation of the behavior of the near field (especially of the clay barrier), is the execution of a very complete experiment, as FEBEX.

Therefore, the FEBEX experiment has the multiple objective of demonstrating the feasibility of fabricating and assembling the engineered barriers and of developing methodologies and models to evaluate the thermo-hydro-mechanical (THM) and thermo-hydro-geochemical (THG) behavior of the near field. These objectives are to be attained with the combination of the "in situ", "mock-up", and laboratory tests. These tests constitute the principal parts of the experiment, according to the program designed by ENRESA.

Fundamentally, the objective of the demonstration is expected to be attained in the "in situ" test, which operates in natural conditions and with components constructed with the actual design dimensions of a repository drift. The demonstration objective has less importance in the "mock-up" test.

The objectives of checking the predictive capacities of the THM and THG numerical models will be supported by the program of laboratory tests designed to study and comprehend the processes that take place in the clay barrier under simple and controlled conditions and to develop the governing equations. The "mock-up" test, at almost full scale and with well-known geometry as well as well-controlled initial and boundary conditions, will serve: to verify the predictive capacity of the coupled numerical models; as a reference for the evaluation, if existing, of the discrepancies; and to adjust the models to predict the behavior of the clay barrier under more heterogeneous conditions, as those in the existing natural system of the "in situ" test.

The underground laboratory managed by NAGRA in Grimsel (Switzerland) has been selected for

the execution of the “in situ” test due to the similarity of the Spanish and Swiss reference concepts for the repository and the suitable laboratory conditions for this test. The “mock-up” test is being executed in the facilities of CIEMAT (Madrid, Spain). The laboratory programs are being performed in various Spanish research facilities (UPC-DIT, CSIC-Zaidín, and CIEMAT).

As was stated above, in the R&D plans of ENRESA in the years prior to FEBEX, a number of sources (deposits) of Spanish bentonite have been studied for use as a clay barrier for a repository. As a result of those studies, the most suitable bentonite was concluded to be that from the deposit called Cortijo de Archidona, which is in the south-eastern part of Spain in the Serrata de Níjar, province of Almería, developed by Minas de Gádor, S.A. Bentonite from this deposit (called sample or bentonite S-2) had been used during the years prior to FEBEX in the investigations of the thermal, hydraulic, mechanical, and geochemical behavior; therefore, extensive information on the properties of this bentonite exists. As a result, it was decided to extract from this deposit the FEBEX bentonite.

From the previous studies, a degree of heterogeneity had been found in the characteristics of the bentonite from this deposit. As a consequence, it was decided to select, homogenize, and process a sufficient quantity (some 300 tons) of bentonite to be used for all of FEBEX—the blocks for the two large-scale tests and the set of experimental laboratory tests—and thereby reduce the problems of evaluating the results from the different tests within this experiment, since it could then be considered that all the bentonite used for the FEBEX project had the same properties. Described within this report are all the pre-operational stage activities, studies, and laboratory tests for selecting, homogenizing, processing, and characterizing the bentonite to be used in FEBEX, from its extraction in the quarry to its final use, including the fabrication,

handling, and storing of the blocks for the construction of the clay barriers of the two large-scale tests. Also included is a summary of the characterization studies made prior to FEBEX, and the results obtained, on samples of bentonite from the same deposit. The laboratory tests referred to herein are those for characterization of the bentonite and should not be confused with the set of experimental laboratory tests being made to investigate the processes and variables.

This report has been divided into seven chapters, with this introduction as Chapter 1. Chapter 2 is a summary and the basic results from the investigations prior to FEBEX. Chapter 3 covers the selection and conditioning of the FEBEX bentonite. In Chapter 4, the characterization of the bentonite prepared for the fabrication of the blocks and for the set of experimental laboratory tests is covered. Chapter 5 describes the activities of design, fabrication, and control of the blocks both for the “in situ” and “mock-up” tests as well as their characteristics before emplacement in their respective sites. In Chapter 6 is a summary of the conclusions reached about the properties of the bentonite as a material and of the blocks fabricated with this bentonite. Chapter 7 is a list of the references referred to herein.

Various institutions, participating in FEBEX, have contributed to this report, including CIEMAT, CSIC-Zaidín, UPC-DIT, and AITEMIN. The report uses the work of CIEMAT as a basis, since they contributed the majority of the testing; however, in each section, the contributions of the other participants have been inserted and credited. (Where no other mention is made of the organization, the contribution is from CIEMAT.) This form of presentation leads to the repetition of some of the names of organizations, but has the advantage of facilitating the comparison and discussion of the results when tests or studies of the same type were performed by two institutions.

2



STUDIES PRIOR TO FEBEX

As has been said in Chapter 1, in the R&D plans prior to FEBEX, ENRESA has studied several Spanish deposits of smectite clays as potential sources for use in constructing a clay barrier for a repository of HLW. Chosen after study was the Cortijo de Archidona, located in Serrata de Níjar (province of Almería, Spain), developed by Minas de Gádor, S. A.

During various years prior to FEBEX, ENRESA had made detailed studies for the characterization and the behavior—mechanical, hydraulic, thermal, and geochemical—of the bentonite from this deposit.

This chapter includes a schematic description of those studies and a detailed summary of the results of the characterization tests made on the Cortijo de Archidona deposit, which became the source of the FEBEX bentonite.

2.1. Study of the potential use of Spanish clays in the engineered barriers

The study of materials to be used for backfilling and sealing a repository for HLW commenced in Spain in 1987, with a project financed by the EU (FI-IW-0191E), participated in by ENRESA, and performed by CIEMAT. That study had three objectives:

- ❑ To survey the availability of Spanish clay suitable as a material for backfilling and sealing.
- ❑ To study the possibility of utilizing an illite clay as an alternative to a smectite clay, in order to minimize the mineralogical differences with the surrounding granite.
- ❑ To study mixtures of clay and ground granite as a material for backfilling in order to, possibly, reduce the costs and contribute to the reconstitution of the surrounding granite environment of a repository.

After surveying the Spanish clays, a study of 30 samples from seven sources located in the provinces of Gerona, Barcelona, Zaragoza, Segovia, Toledo, Jaén, and Almería was initiated. The first semiquantitative determination of the mineralogy excluded 50 % of the samples due to the low content of phyllosilicates (<65 %). In accordance with the mineralogy of the fraction less than 2 μm , seven of the samples were retained: two were essentially illite, the rest were smectites provided by

Minas de Gádor from the region of Cabo de Gata (Almería) and from the Tertiary Madrid Basin (Yuncos, Toledo). Finally, among those, two clays were selected for their low content of carbonates and colloidal minerals, their high plasticity, and their good compactability: one a bentonite called “Serrata natural” from the deposit of Cortijo de Archidona (Cabo de Gata, Almería) and the other an illite from Zaragoza. The analyses of the mechanical, hydraulic, thermal, and physico-chemical properties of both and their behavior when mixed with ground granite [6] led to the following conclusions:

- ❑ Spanish clays exist which could be utilized as material for backfilling and sealing.
- ❑ The smectite clay is more suitable than the illite clay as a material for backfilling and sealing.
- ❑ The ground granite mixed with smectite clay can be used as material for backfilling, provided that the ground granite does not exceed 25 % of the mixture.

2.2. Study of Cabo de Gata and tertiary Madrid basin zones

Partially coinciding with the development of the study described above, ENRESA promoted the study of two zones with important deposits of smectite clays that could be utilized as materials of backfilling and sealing: Cabo de Gata in the province of Almería and the Tertiary Madrid Basin in the province of Toledo. In each of these zones, three deposits were considered as potential sources due to their characteristics (which were already known) and their estimated reserves. For the zone of Cabo de Gata, the deposits selected were Cortijo de Archidona (in the Serrata de Níjar), Morrón de Mateo (in the Rambla de los Escullos), and Los Trancos (in the northern zone of Sierra de Gata); for the zone of the Tertiary Madrid Basin, the deposits were Cerro del Águila-Cerro del Monte, Santa Bárbara, and Yuncos. Each of these deposits were studied in detail by CIEMAT, with CSIC-Zaidín, for the zone of Cabo de Gata, and with Universidad Autónoma de Madrid (UAM), for the zone of Madrid Basin. In addition, CIEMAT had the collaboration of the Commissariat à l’Energie Atomique (CEA).

During these studies, ENRESA had the assistance of R. Pusch of Clay Technology (Sweden).

CIEMAT received two samples from each deposit, each about 200 kg. The grain-size distribution, Atterberg limits, specific weight, total and external specific surfaces, and mineralogical and chemical composition were determined for each sample. Also compaction tests, both dynamic and static, were made and, in the CEA laboratory, the surface thermal conductivity was determined in compacted samples [7, 8, 9].

CSIC-Zaidín (for the Cabo de Gata zone) and UAM (for the Madrid Basin zone) made complete characterization, mineralogical and chemical, as well as studied the alterability and stability of various samples from the same deposits. The results obtained were collected in the reports published by ENRESA and by the EC [10, 11, 12].

Using the results from all the laboratories, one deposit from each zone was chosen: Cortijo de Archidona (formed essentially of smectite of the montmorillonite type) in Cabo de Gata and Cerro del Águila-Cerro del Monte (consisting fundamentally of trioctahedral smectite of the saponite type, accompanied by various proportions of illite and sepiolite) in Tertiary Madrid Basin. These deposits were selected because they met the requirements for the material to backfill and seal a repository for HLW: mineralogical purity, high specific surface, high liquid limit, high percentage of the fraction less than 2 μm , and high thermal conductivity; furthermore, good compactability was an added requirement for the fabrication of the highly-compacted blocks for the clay barrier [7].

Selection of the Cortijo de Archidona (Cabo de Gata) deposit

A more specific investigation of the two selected deposits was continued by CIEMAT to determine the more suitable one from which to obtain the material for backfilling and sealing a repository. After this study, the Cortijo de Archidona, located in the Serrata de Níjar (zone of Cabo de Gata, Almería) was chosen.

For these selection studies, CIEMAT acquired a sample from each deposit. Both samples, each of 5000 kg, were prepared in the facilities of the company Minas de Gádor by drying at 60°C until the water content was approximately 10% and by grinding until the maximum size was 5 mm.

The sample from Cortijo de Archidona was designated S-2 and it is with this designation that all information (prior to FEBEX) relating to the study of

the smectite clay (bentonite) from this deposit was published. The sample from the Cerro del Águila-Cerro del Monte deposit was designated MCA-C.

The clays were ground to a maximum size of 2 mm (except in those cases where the effect of the grain-size distribution was specifically investigated) and they were stabilized at the temperature and moisture of the laboratory (20°C to 25°C and relative humidity about 60%), prior to the execution of the tests.

The study made by CIEMAT included not only tests for determining the properties of the clays, but also for investigating the variations in the properties as a function of the dry density and water content of the compacted blocks, of the temperature during the test, of the grain-size distribution, of the addition of quartz sand in different proportions, and of the pre-heating of the clay to different temperatures. On samples from both deposits, the following tests were made: dynamic compaction, unconfined compressive strength, free swelling, swelling pressure, saturated hydraulic conductivity, thermal conductivity, consolidation, and consolidated triaxial (drained and undrained). Furthermore, the isotherms of adsorption/desorption were determined and studies were made of ionic diffusion and hydrothermal alterability. In addition, the alterations, from heating, on the mineralogy; specific weight; external specific surface; and texture, observed under an electronic microscope, were determined. Simultaneously, under contract with CEA, uniaxial compaction tests of industrial scale were made with clays from both deposits in collaboration with Constructions Thermiques Européennes (Montsempron-Libos, France).

These studies and their results have been published [7,13,14,15,16]. The results led to the definitive selection of the bentonite from Cortijo de Archidona, designated S-2, for its better compactability and lesser susceptibility to thermal effects, although both clays satisfy the requirements for utilization as material for the clay barrier. It was concluded that a dry density of 1.6 g/cm³ satisfied the hydraulic requirements for the clay barrier and that the addition of quartz sand did not greatly improve the properties of the material.

The evaluation of the fabrication and construction of the clay barrier to be made of the bentonite S-2 (Cortijo de Archidona) was by collaboration among ENRESA, CIEMAT, and CEA. The mold and the fabrication system for the compacted clay blocks, as well as the method of installation, were designed in the manner employed by the French.

Their corresponding “in situ” investigative test was made in drilled wells, with a diameter of 1.50 m and a depth of 6.00 m, in the old mine of Cogema (Fanay-Silord, France) [12].

2.3. Longevity studies

For the long-term performance of a repository, the integrity of the clay barrier is fundamental; thus, the changes that the clay undergoes during the lifetime of a repository must be studied. The changes are controlled by the thermal impact, the hydration water, and the chemical environment. An important change that the clay barrier can undergo is the possible transformation of montmorillonite to illite with the resulting deterioration of its mechanical, swelling, and retention properties.

In the period 1992-1995 in CSIC-Zaidín, UAM, and CIEMAT laboratories the materials have been tested at various concentrations of salinity, temperatures, and time intervals to determine the reaction kinetics. CSIC-Zaidín has established an equation that governs the transformation of montmorillonite to illite; the equation indicates, that at a temperature of less than 120°C, the transformation takes place during hundreds of thousands to millions of years, depending on the quantity of potassium available in the environment [17]. The studies from UAM did not find any relevant transformation that would affect the saponite for temperatures under 200°C, but did establish kinetic equations that control the transformation of the accessory minerals (sepiolite) into saponite and that indicate the transformation is produced in decades.

CSIC-Barcelona has studied the bentonite of Almería to obtain thermodynamic and kinetic information on the process of dissolution/precipitation.

2.4. Study of THM behavior of the bentonite S-2 (1991-1995)

In 1991, another study of the bentonite S-2, from the deposit of Cortijo de Archidona, was initiated. This study was part of the scope of the “Anexo V del Convenio de Asociación CIEMAT-ENRESA Investigación y desarrollo tecnológico en el campo de la migración de radionucleidos y comportamiento de barreras naturales y artificiales frente a la migración” in its Annex C: “Estudios básicos de comportamiento térmico, hidráulico y mecánico de materiales de barreras artificiales”. For this study,

24 tons of material, provided by Minas de Gádor in October 1990, was prepared in their plant in Almería by drying at 60°C and grinding to obtain a maximum size of 5 mm. For its use in the laboratory of CIEMAT, the material was again ground to a maximum size of 2 mm.

The object of the study was to analyze the properties of the bentonite and its behavior under conditions that will be found in a repository; that is to say, to subject the material simultaneously to heating and hydration, in opposite directions, for long periods of time. This includes the study of the effects produced by the thermal and hydraulic flows in the compacted blocks and the behavior of the material in subsaturated conditions. Furthermore, the characterization of the bentonite was continued, undertaking new aspects like the determination of the pore size distribution and the specific heat as a function of the temperature, and additional determinations of specific weight, cation exchange capacity, exchangeable cations, thermal conductivity, etc. Other aspects undertaken for the first time were the extraction and analysis of the interstitial water and soluble anions as well as the bacteriological analyses.

2.5. Summary of characterization tests for bentonite S-2, prior to FEBEX

The properties of the bentonite S-2 from the Cortijo de Archidona deposit, obtained in the studies that have been described in the two previous sections, are briefly summarized in this section.

2.5.1. Origin

During the Neogene, the Cabo de Gata zone suffered an intense volcanic activity, which is included within the geotectonic frame of the western Mediterranean. These volcanic rocks were subjected to a process of transformation caused by hydrothermal fluids that circulated due to the great density of fractures. It is thought that the bentonite of the Cortijo de Archidona deposit had originated from polygenetic tuffs and porphyritic agglomerates of dacitic character, that suffered an initial process of devitrification by hydrothermalism (at temperatures between 75°C and 100°C) and later had been altered (at temperatures around 40°C) by the action of meteoric water infiltrated through the fractures [18].

2.5.2. Mineralogical and chemical composition

The bentonite S-2 is essentially smectite ($92\pm4\%$), with cristobalite, quartz, plagioclase, and calcite, each in proportions less than 5%. The fraction less than $2\text{ }\mu\text{m}$ is formed mainly by dioctahedral smectites of montmorillonite type and its structural formula is shown in Table 2.1.

The chemical analyses of the total sample, including the trace elements, performed by CSIC-Zaidín [10] and CIEMAT [13] are consistent and are shown in Table 2.2.

The cation exchange capacity and exchangeable cations were determined in both laboratories and are summarized in Table 2.3.

In addition, the exchange complex includes Sr, Ba, Mn, and occasionally Al.

The contents of amorphous phases are indicated in Table 2.4. The procedure for attacking the colloidal silica in the laboratory of CSIC-Zaidín [10] is more intense than that utilized in CIEMAT, which explains the great difference observed in the values from the two laboratories.

The characteristics of the aqueous extracts in the solid to liquid relation 1:4 (CIEMAT) and 1:10 (CSIC-Zaidín) are shown in Table 2.5.

2.5.3. Identification properties

The identification properties of the bentonite S-2 are summarized in Table 2.6 and include unpublished values obtained by CIEMAT and CSIC-Zaidín in the years prior to FEBEX.

Determination of the grain-size distribution of this clay presents complications due to the difficulty in fully dispersing the particles in a suspension. This difficulty has been attributed to the binding or cementation produced by colloidal sil-

ica [9,10]. CSIC-Zaidín utilized Na_2CO_3 and cycles of ultrasound and CIEMAT used sodium hexameta-phosphate with cycles of ultrasound and stirring to disperse the material.

2.5.4. Strength parameters

Unconfined compressive strength

The unconfined compressive strength tests were made on cylindrical specimens of 38 mm in diameter and 78 mm in height, with a loading rate of 2 % of the deformation per minute. The influence of the compaction system (tamping or uniaxial), the water content, the dry density of fabrication, the ground grain-size distribution, and the addition of quartz sand in different proportions were analyzed. It was shown that the unconfined compressive strength increased in an exponential manner with an increase in dry density and that the unconfined compressive strength was 25 kg/cm^2 for a dry density of 1.70 g/cm^3 with the humidity at equilibrium with the conditions in the laboratory. The higher strengths were obtained with water contents of about 20 %. The increase in ground grain size and the addition of quartz sand caused a decrease in the unconfined compressive strength.

Triaxial tests

The strength parameters obtained in the consolidated triaxial tests, both drained (CD) and undrained (CU), made on saturated specimens with an initial dry density of 1.60 g/cm^3 are described in [7] and summarized in Table 2.7. In the CD tests, the failure is produced with strains between 7% and 14% by values of the deviator stress of 1.7 MPa and 2.7 MPa. In the CU tests, the strain at failure is between 9% and 12% for values of deviator stress between 1.5 MPa and 2.9 MPa and between 8%

Table 2.1
Structural formula of the smectite S-2

	Tetrahedral		Octahedral			
	Si ⁴⁺	Al ³⁺	Al ³⁺	Fe ³⁺ , Fe ²⁺	Mg ²⁺	Ti ⁺
CSIC- Zaidín [10]	7.76±0.10	0.24±0.10	2.79±0.05	0.33±0.05	1.03±0.10	
CIEMAT [13]	7.97	0.03	2.74	0.43	0.89	0.02

Table 2.2
Chemical composition of the bentonite S-2 total sample, including trace elements

Oxides %	CSIC-Zaidín	CIEMAT	Trace elements ppm	CSIC-Zaidín	CIEMAT
SiO ₂	59.9±1.7	57.3	B	53±38	
Al ₂ O ₃	18.7±1.1	19.6	Ba	110±63	195
Fe ₂ O ₃	3.6±0.4	3.9	Be		<5
FeO		0.4	Cl ⁻		552
MnO		<0.03	Co		5.3
MgO	5.8±0.4	4.7	Cr	15±11	15
CaO	2.0±1.1	2.9	Cu	5±2	<5
Na ₂ O	2.2±0.2	1.9	La		26
K ₂ O	0.7±0.3	1.4	Mo	7±3	<5
TiO ₂		0.3	Ni	16±5	20
P ₂ O ₅		0.1	Pb	41±22	
H ₂ O ⁺	6.7±0.9	5.7	Rb		52
CO ₂ org.	0.7±0.3	0.6	Sn	41±30	
CO ₂ min.		0.1	Sr		210
SO ₂ (S ²⁻)		0.02	Th		15.7
SO ₄ ²⁻		0.03	U		1.02
F ⁻		0.16	V	11±13	17
			W		<25
			Y		23
			Zn		77
			Zr	337±320	180

Table 2.3
Cation exchange capacity and exchangeable cations of the bentonite S-2, in meq/100 g

	CEC	Ca ²⁺	Mg ²⁺	Na ⁺	K ⁺
CIEMAT [19]	97±17	41±7	31±7	22±3	2.5±0.4
CSIC-Zaidín [10]	92±12	31±6	33±6	30±3	3±1

Table 2.4
Content of amorphous silica, alumina, and iron of the bentonite S-2

	SiO ₂	Al ₂ O ₃	Fe ₂ O ₃
CSIC, in %	1.92±1.38	0.09±0.05	0.11±0.02
CIEMAT, in %	0.025±0.01		

Table 2.5
Analysis of aqueous extracts of the bentonite S-2

	Aqueous extract 1:4 CIEMAT	Aqueous extract 1:10 CSIC-Zaidín
Cl ⁻ , in mg/l	299±75	
SO ₄ ²⁻ , in mg/l	314±38	
E.C., in mS/cm	1.884±0.241	0.792±0.151
pH		8.57±0.43

and 11% for values of the deviator stress between 2.4 MPa and 6.6 MPa.

Triaxial tests were made with material in an unsaturated state and their results are shown in Tables 2.8 and 2.9. For the lower ranges of σ_3 , the failure is brittle, is produced for strains under 8%, and has a strength peak, whereas for the higher ranges of σ_3 , the failure is plastic and is produced for strains on the order of 20%.

2.5.5. Oedometer tests

The oedometer tests were made with loading and unloading cycles on specimens compacted to an initial dry density of 1.4 g/cm³ and 1.6 g/cm³. To analyze the results, the Terzaghi theory of consolidation was applied and the curves analyzed by the empirical procedure of Casagrande. The results are shown in Table 2.10.

2.5.6. Oedometer tests with controlled suction

The oedometer tests with controlled suction were made at 20°C by dissolutions of sulfuric acid or by nitrogen pressure [20, 21, 22]. The tests have been made on specimens compacted to a dry den-

sity of 1.6 g/cm³, with a water content at equilibrium with laboratory conditions. By fitting the experimental data, the following preliminary state surface is obtained [23]:

$$e = 3.642 - 0.197 \ln p - 0.13 \ln s + 0.009 \ln p \ln s$$

where e is the void ratio, s is the suction in MPa, and p is the net mean stress measured in MPa.

2.5.7. Swelling pressure

The swelling pressure has been determined in an oedometer, where the specimen is saturated at constant volume by adding successive loads. Mixtures of clay and quartz sand were used with dry densities between 1.3 g/cm³ and 2.0 g/cm³ for the clay. The fitted curve is shown in Figure 2.1 and given by the equation

$$\ln P_s = 5.9 \rho_d - 7.9 \quad r^2 = 0.93 \quad 17 \text{ points}$$

where P_s is the swelling pressure in MPa and ρ_d is the dry density of the clay in g/cm³ [24].

2.5.8. Saturated hydraulic conductivity

The saturated hydraulic conductivity of specimens of distinct dry density has been determined, by Darcy’s law, from tests in which a hydraulic gradient was applied, between the two ends of a specimen of known dimensions, and the volume of water that traversed the specimen was measured as a function of time. The logarithm of the values obtained for the hydraulic conductivity (k , in m/s) as a function of the dry density (ρ_d , in g/cm³) is represented in Figure 2.2. As can be observed, the val-

ues have been fitted to two straight lines depending on the interval of the dry density being considered [24]. The regression lines are expressed by the equations

for ρ_d between 1.4 and 1.9 g/cm³:
 $\log k = -2.97 \rho_d - 8.29 \text{ (} r^2=0.80 \text{)} \quad 9 \text{ points}$

for ρ_d between 1.1 and 1.4 g/cm³:
 $\log k = -8.14 \rho_d - 0.72 \text{ (} r^2=0.88 \text{)} \quad 12 \text{ points}$

Table 2.6
Identification properties of the bentonite S-2

Property	Laboratory	
	CIEMAT	CSIC-Zaidín
Water content in equilibrium with laboratory conditions, in %	10 to 13	
Liquid limit, in %	215±55 (*)	
	105±10 (**)	
Specific weight	2.78	
Grain-size distribution, in %:	<2µm	82±6
	<74µm	93±3
Total specific surface, in m²/g	516±37	614±74
External specific surface BET, in m²/g	37	

(*) It is the value obtained on the first samples from the deposit.
(**) It is the value obtained on samples from the deposit, taken years later.

Table 2.7
Table 2.7: Strength parameters from saturated triaxial tests $\rho_d=1.60 \text{ g/cm}^3$ (bentonite S-2)

Type of test	Range of σ_3 MPa	Cohesion MPa	Friction angle in degrees
CD	3-10	0.0	7
CD	10-30	0.2	4
CU	3-10	0.2	8
CU	10-30	0.2	5

Table 2.8:
Strength parameters from non-saturated triaxial tests $\rho_d=1.65\text{ g/cm}^3$ and different water contents (bentonite S-2)

w %	Initial S_r %	Range of σ_3 MPa	Cohesion MPa	Friction angle in degrees
10	41	0.5-3.0	0.1	33
10	41	10.0-30.0	7.0	8
15	61	0.5-3.0	0.8	31
20	81	0.5-3.0	1.2	21
20	81	10.0-30.0	3.0	2

Table 2.9
Strength parameters from non-saturated triaxial tests, with the water content at laboratory equilibrium and with different initial densities (bentonite S-2)

ρ_d g/cm ³	Initial S_r %	Range of σ_3 MPa	Cohesion MPa	Friction angle in degrees
1.6	41 - 47	0.5-3.0	0.7	25
1.6	41 - 47	3.0-10.0	2.8	14
1.6	41 - 47	10.0-30.0	4.4	14
1.7	49 - 60	0.5-3.0	0.8	30
1.7	49 - 60	3.0-10.0	1.0	26
1.7	49 - 60	10.0-30.0	3.5	16
1.8	53 - 59	3.0-10.0	4.3	16
1.8	53 - 59	10.0-30.0	3.6	18
1.9	65 - 79	3.0-10.0	4.5	19

Table 2.10
Oedometric parameters. Bentonite S-2

ρ_d g/cm ³	C_c	C_s	a_v m ² /kN	m_v m ² /kN	k m/s
1.4	0.38	0.20	$2.5 \cdot 10^{-5}$	$1.7 \cdot 10^{-5}$	$4.7 \cdot 10^{-12}$
1.6	0.38	0.33	$2.3 \cdot 10^{-5}$	$1.4 \cdot 10^{-5}$	$1.3 \cdot 10^{-13}$

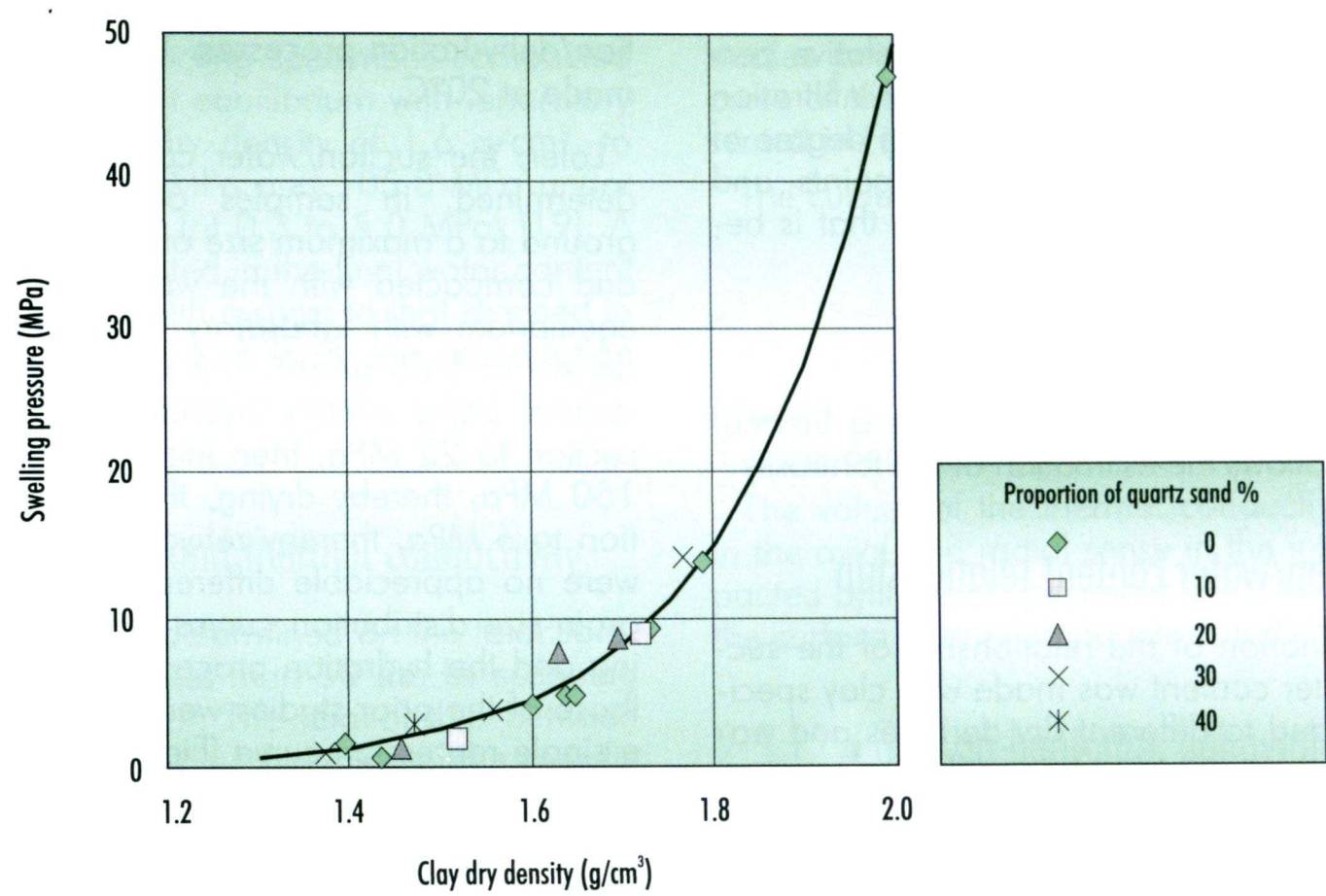


Figure 2.1: Experimental values and regression curve for the swelling pressure vs. dry density for bentonite S-2 mixed with different proportions of quartz sand.

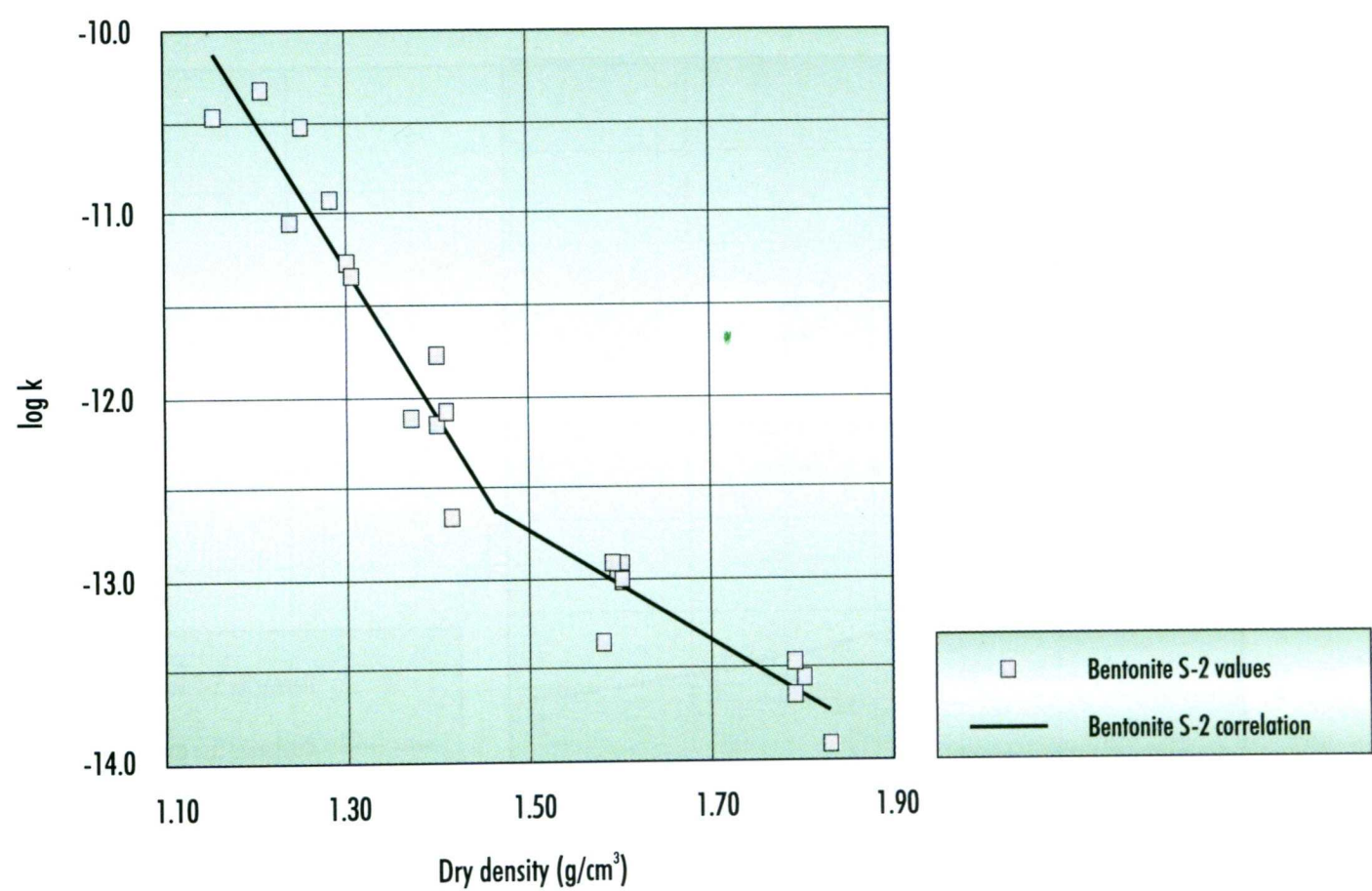


Figura 2.2: Experimental values and regression straight lines for the logarithm of the hydraulic conductivity (k , in m/s) vs. dry density (ρ_d , in g/cm^3) for the bentonite S-2.

2.5.9. Relative permeability

UPC-DIT has adopted a law for the relative permeability (k_{rl}) from the inverse analysis of infiltration test data from CIEMAT [24], in which the degree of saturation (S_r) is known at different points and times in a specimen of compacted clay that is being saturated: the law obtained is

$$k_{rl} = S_r^3$$

The performance of these tests under a thermal gradient also allows the estimation of the tortuosity.

2.5.10. Suction/water content relationship

The determination of the relationship of the suction to the water content was made with clay specimens compacted to different dry densities and water contents, then subjected to different suctions (greater or less than the initial one in the specimen) until the water content stabilized in the clay [20]. The suction was imposed by controlling the relative humidity of the atmosphere within the specimen container by dissolutions of differing densities of sulfuric acid or by applying the axis-translation technique in membrane cells. In both cases, the

volume of the specimen (and therefore the void ratio) varied without restraint during the hydration/dehydration processes. The determination was made at 20°C.

Later, the suction/water content curve was also determined, in samples of the bentonite S-2 ground to a maximum size of 2 mm, and of 5 mm, and compacted with the water content (10%) at equilibrium with laboratory conditions and a dry density of 1.65 g/cm³. This was made following a suction path, initially hydrating by reducing the suction to 22 MPa, then increasing the suction to 150 MPa, thereby drying, then reducing the suction to 6 MPa, thereby rehydrating. Because there were no appreciable differences between the two grain-size distribution curves nor between the drying and the hydration processes, these results and those of the prior studies were integrated to obtain a single regression curve (Figure 2.3). The relation obtained between suction (s , in MPa) and water content (w , in %) that is independent of both the initial dry density and initial water content is

$$w=36.1-12.0 \log s \quad (r^2=0.94) \quad 191 \text{ points}$$

Various tests were made in an oedometer, at 20°C, with controlled suction, to identify, in the

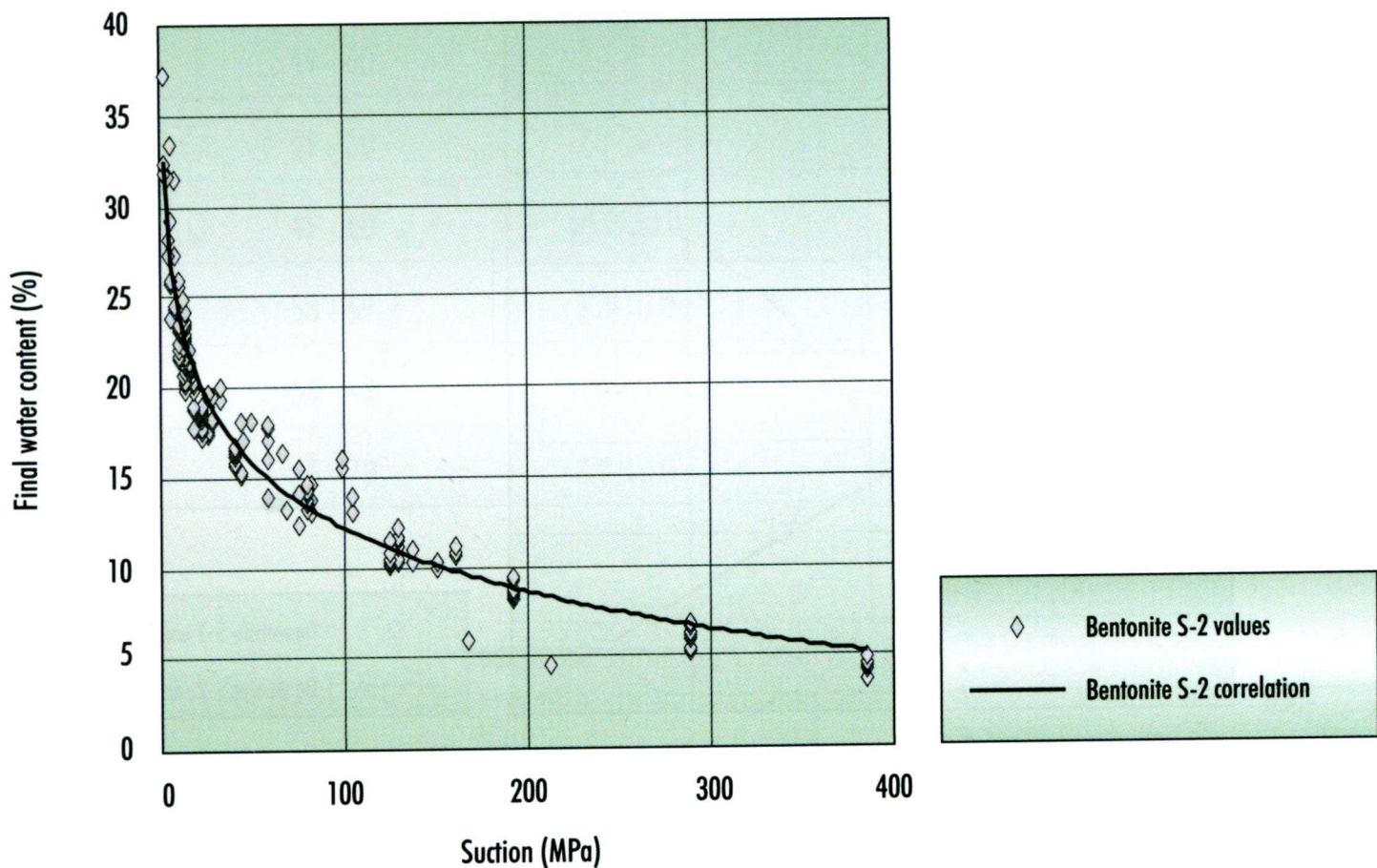


Figure 2.3: Suction/water content curve for bentonite S-2

suction/water content relationship, the influence of a load to which a specimen has been subjected. This consisted of subjecting specimens compacted with a water content at equilibrium with laboratory conditions and at a dry density of 1.6 g/cm³, to suctions between 0.3 MPa and 105.0 MPa under different vertical loads (of 0.3 to 5.0 MPa) [19]. A slight decrease was noted in the final water content for the same suction with respect to that reached in specimens not subjected to load, but no variation was observed as a function of the magnitude of the applied load.

2.5.11. Specific heat and thermal conductivity

The specific heat is determined on the total sample ground to a size less than 50 μm by scanning differential calorimetry. The relationship between temperature (T, in °C) and the value of the specific heat of the solid (c_s, in J/kg · °C), in an interval of temperature between 45°C and 150°C, is adjusted to a straight line equation

$$c_s = 1.38 T + 732.52$$

The surface thermal conductivity has been determined at ambient temperature on compacted clay

specimens of different water contents and different dry densities. The values obtained in the latest studies prior to FEBEX, together with a regression curve of the sigmoidal type (Boltzmann), are represented in Figure 2.4.

The curve obtained corresponds to the equation

$$\lambda = 1.34 - \frac{0.95}{1 + e^{(S_r - 0.54)/0.15}}$$

where λ is the thermal conductivity in W/m·K and S_r is the degree of saturation.

The values of the thermal conductivity measured in the axial and radial sense in the interior of compacted cylindrical specimens are similar to those of the surface thermal conductivity indicated [26].

2.5.12. Effect on bentonite behavior of the maximum grain size

Since the major part of the characterization tests on the S-2 bentonite were made with the material ground to a maximum grain size of 2 mm, and as the bentonite to be utilized in FEBEX was to be ground to a maximum size of 5 mm, it was consid-

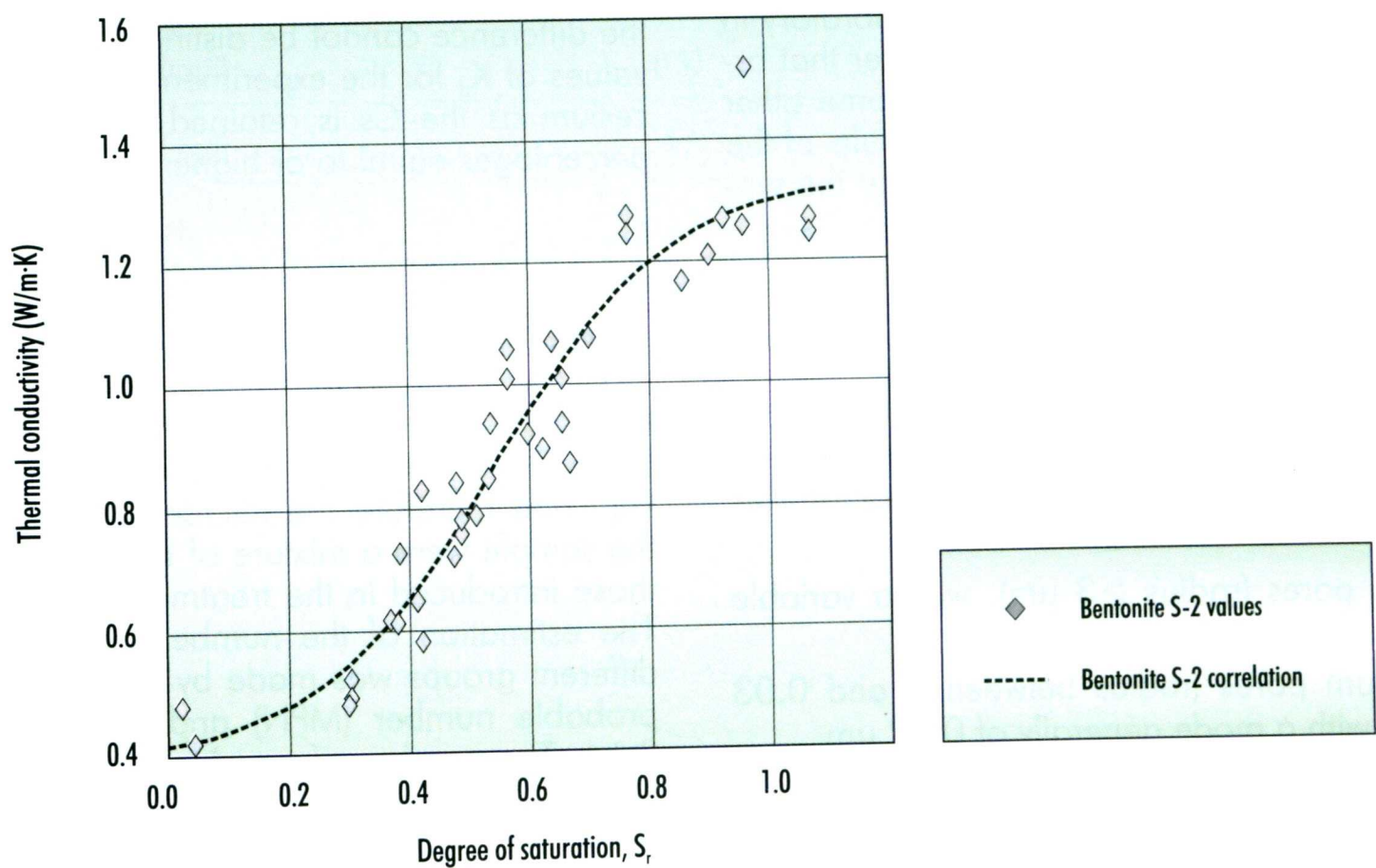


Figure 2.4: Thermal conductivity of the bentonite S-2 as a function of the degree of saturation.

ered necessary to make a set of tests to verify that the thermal, hydraulic, and mechanical behavior were unaffected by the grain-size distribution of the ground material [19]. Swelling under load tests, hydraulic conductivity, thermal conductivity, and porosity determinations, with dry densities between 1.4 g/cm³ and 1.8 g/cm³ were made. Also, for the two grain-size distributions, the suction/water content curve was determined and oedometer tests with controlled suction were made. In no tests were differences found with respect to the grain-size distribution. The infiltration curve in tests made in thermo-hydraulic cells have similar tendencies regardless of the maximum grain size of the ground clay [25].

2.5.13. Interstitial water

The interstitial water of the clay has been obtained by compaction, under a pressure of 70 MPa, of a clay paste with a water content of 25 % obtained by mixing the clay, stabilized under the laboratory conditions, and granite water in a necessary proportion. Various tests have shown that the chemistry of the water obtained is conditioned by the clay and not by the type of water added [27]. The water obtained is of a chloride-sodic-magnesian type, with an ionic strength of 0.3 M. From these analyses, a procedure has been developed for the preparation of a stable synthetic bentonite-granite water for use in the laboratory in this and other studies to simulate the water that arrives at a point after passage through some other layer of bentonite [28]. The analytical results of the extracted water and the allowable limits for the synthetic water are shown in Table 2.11.

2.5.14. Porosity

From the study, by the mercury intrusion method, of the compacted clay specimens at different densities and with different water contents, three families of pores have been identified:

- large pores (radius >3 μm), with a variable mode
- medium pores (radius between 3 and 0.03 μm), with a mode generally of 0.32 μm
- small pores (radius between 0.03 μm and 0.003 μm), with a mode generally of 0.006 μm

The percentage of small pores is always greater than that of the medium pores, whereas the per-

centage of large pores is quite variable and is inversely related to the compacted density of the clay.

2.5.15. Sorption of cesium

By the batch technique of testing, a study was made of the adsorption of ¹³⁷Cs into the bentonite S-2 ground to a grain size of less than 63 μm. These tests were made considering different parameters for determining their influence on the process of retention: time, initial pH (6 to 9), initial ionic strength (0.004 M to 0.100 M), and cesium concentration (10⁻³ M to 10⁻⁹ M). The tests were effected in an oxic environment. The detailed description can be found in [29].

In all of the tests, it was observed that the S-2 bentonite presented an important buffer effect or regulator of pH. The cesium retention capacity of the bentonite increases with increasing pH, with the decrease of the cesium concentration, and with the decrease in the ionic strength. In general, there is not a notable variation in the value of the distribution coefficient (K_d) with time.

The values obtained for the distribution coefficient in the tests made with an ionic strength of 0.004 M are grouped together in Table 2.12.

Table 2.13 shows the results of the tests made with ionic strengths of 0.01 M and 0.10 M, where the difference cannot be distinguished between the values of K_d for the experimental concentrations of cesium as the Cs is retained by the bentonite in percentages equal to or higher than 98 %.

2.5.16. Bacteriological analysis

Only one preliminary bacteriological study was made on a sample of the S-2 bentonite [30]. In the extraction of the sample, as in the later treatment (drying and grinding), sterile conditions were not kept and therefore the microorganisms present in the sample were a mixture of the natural ones and those introduced in the treatment of the bentonite. The estimation of the number of bacteria of the different groups was made by the method of most probable number (MPN) and is shown in Table 2.14. The number of aerobic and sulfide oxidizing bacteria were greater than expected, even though the numbers could not be correctly estimated.

Only analyses of the presence or absence of some groups of bacteria were made without making an estimate of the number.

Table 2.11
Chemical composition of the bentonite S-2 interstitial water

	Interstitial water	Synthetic water
Cl ⁻ (mg/l)	6570	6550±250
NO ₃ ⁻ (mg/l)	67	110±10
SO ₄ ²⁻ (mg/l)	1490	1500±30
HCO ₃ ⁻ (mg/l)	170	27±5
SiO ₂ (mg/l)	12	8.3±0.5
Br ⁻ (mg/l)	16	15±1
Ca ²⁺ (mg/l)	575	135±10
K ⁺ (mg/l)	11	21±1
Mg ²⁺ (mg/l)	1 100	600±30
Na ⁺ (mg/l)	2425	3750±100
Mo (mg/l)	0.13	
Sr (mg/l)	12	
B (mg/l)	0.38	
Ni (mg/l)	0.73	
Cu (mg/l)	0.37	
Zn (mg/l)	4.8	
pH	7.2	7.3±0.2
E.C. (mS/cm)	18	18.5±0.2
Eh (mV)		430±30

Table 2.12
Values of K_d (ml/g) as a function of the pH and the concentration of Cs in tests with an ionic strength of 0.004 M. Bentonite S-2

Final pH	[Cs] = 10^{-3} M	[Cs] = 10^{-5} M	[Cs] = $10^{-7} - 10^{-9}$ M
7.25	425 ± 20	910 ± 155	11550 ± 290
↓	485 ± 15	975 ± 190	↓
	560 ± 15	1270 ± 220	
	730 ± 70	1390 ± 290	
8.34			15000 ± 1120

Table 2.13
Values of K_d (ml/g) from tests with ionic strengths of 0.01 M and 0.10 M. Bentonite S-2

[Cs] = $10^{-7} - 10^{-9}$ M	
Ionic strength 0.01 M	9400 – 11600
Ionic strength 0.1 M	4700 – 5800

Table 2.14
Preliminary bacteriological analysis of the bentonite S-2

Type of bacteria	Most Probable Number (MPN) per gram
Aerobic	> 2500
Anaerobic	+
Sulfur oxidizing	> 2500
Sulfate reducing	+

The symbol + indicates presence of bacteria.

3

SELECTION AND CONDITIONING OF THE FEBEX BENTONITE

3.1. Selection

From the studies prior to FEBEX, as was stated in Chapter 2, the bentonite from the deposit called Cortijo de Archidona (Serrata de Níjar, Almería), developed by Minas de Gádor, S. A., was selected as the reference material for use in backfilling and sealing a repository for high-level radioactive waste. Consequently, it was decided to utilize the bentonite from this deposit for the FEBEX project.

Given the variation in colors and textures that had been observed in the bentonite deposit, it was decided to make a detailed investigation of a zone within the quarry from which the required quantity (some 300 tons) of bentonite would be extracted and conditioned in order that it have the homogeneity and properties required for the FEBEX project. After the investigation, Minas de Gádor proposed the use of an existing storage pile of some 900 tons of rose-colored bentonite that had been extracted from the zone being investigated. After analyses, it was decided to utilize this pile of bentonite as it met the technical characteristics required and precluded the need for further excavation.

In the report 70-IMA-L-3-1 *Selección, homogeneización y análisis de una partida de bentonita procedente de la cantera "La Serrata" (Almería) para el proyecto FEBEX* [31], the complete process of selecting and conditioning of the bentonite is described in detail.

3.1.1. Characteristics of the quarry

The bentonite deposit is situated in a zone which has been affected by the Cenozoic volcanism of the andesite-dacite type. The majority of the volcanic material has had epigenetic alteration after the volcanism, of a pneumatolytic and hydrothermal nature related to a great density of fractures.

The sides of the quarry have been excavated in barren volcanic rocks with the bentonite outcropping at its base and the better material found in the central section. In general, the deposit has the following characteristics:

- The presence of bentonite coincides with the direction of the tension faults within the shear fault zone of La Serrata. The plane of the fault of the bentonite deposit is subvertical, dipping to the north or to the south.
- The fault marks the limit between the overhead roof rock and the bentonite.

- The overhead roof rock is compact rhyodacite of light gray color.
- The underhand wall rock is volcanic tuff formed by abundant volcanic fragments of different materials.
- The composition of the host rock is of great importance as there appears to be a correspondence between the type of host rock and the quality of the bentonite.
- The bentonite has less impurities close to the overhead roof rock and center, where the white and rose-colored materials predominate, whereas the bentonite in the underhand wall rock has more impurities.
- From the position of the bentonite it can be deduced that the fractures favored the circulation of fluids that hydrothermally alter the rock.

Figure 3.1 shows schematically the quarry.

3.1.2. Sampling of the bentonite from the quarry and storage pile

Based on the characteristics of the quarry, a zone of 1800 m² was selected in the center (Figure 3.1) on which were marked six profiles (P0 to P5), in a north-south direction, and where, in test pits, 22 samples of approximately 2 kg each were taken and designated C-0.1 to C-0.7, C-1.1 to C-1.5, C-2.1 to C-2.3, C-3.1 to C-3.3, C-4.1 to C-4.3, and C-5.2. (The letter C designates the quarry; the first number, the profile from which the sample was taken; and the second number, the test pit within the profile, as is explained in Procedure 70-IMA-G-3-70 [32].) In a larger test pit, centrally located in the zone being investigated, 5 other samples were taken and designated CC0-1 to CC0-5; another one, designated C-A, was taken between profiles 1 and 2.

To evaluate the characteristics of the bentonite in the existing storage pile, 8 samples were taken along the surface and they were designated PI-1 to PI-8.

3.1.3. Laboratory tests

The properties of the bentonite have been determined by laboratory tests performed by CIEMAT on samples obtained as indicated above.

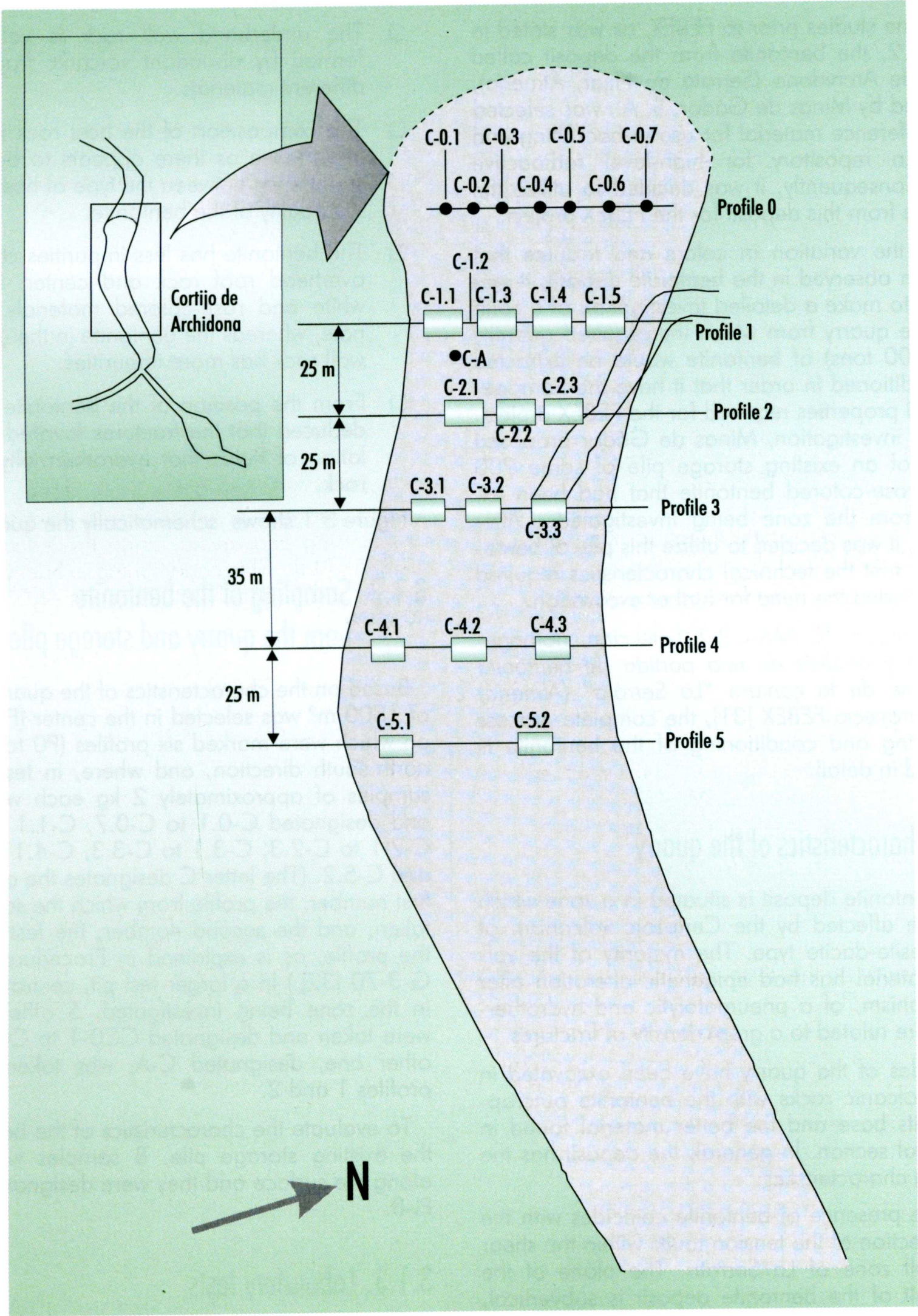


Figure 3.1: Cortijo de Archidona scheme, profiles, and sampling test pit locations.

3.1.3.1. Sample preparation

Before testing, the samples were prepared in accordance with Procedures 70-IMA-G-3-70 [32] and 70-IMA-G-3-71 [33] using these processes:

- ❑ Spreading out and air-drying of samples C-2.1, C-3.2, and C-4.3 and oven-drying of the rest of the samples at 60°C for 48 hours.
- ❑ Crushing of the clods with a rubber mallet and wooden roller.
- ❑ Sieving the samples to determine the percentage of particles less than 5 mm, after the sample has been dried and the clods crushed.

3.1.3.2. Testing procedures

On the fraction of the material smaller than 5 mm, the following tests were made using the procedures indicated:

- ❑ Water content determination: standard UNE 103-300-93 [34].
- ❑ Grain-size determination by sieving to the 74 μm : Procedure 70-IMA-G-0-05 [35].
- ❑ Atterberg limits: Procedure 70-IMA-G-0-12 [36]
- ❑ Swelling pressure of uniaxially compacted samples with a dry density of 1.60 g/cm³ and the water content that was reached after drying: Procedure 70-IMA-G-0-21 [37].
- ❑ External specific surface, BET: Procedure 70-IMA-G-0-11 [38].

3.1.3.3. Test results and bentonite selection

The results of the tests are included in Tables 3.1, 3.2, and 3.3.

The following three specifications (Procedure 70-IMA-G-70 [32]) were established for the selection of the bentonite:

- ❑ The fraction of the particles larger than 5 mm must be less than 5 % and the fraction of particles less than 74 μm must be more than 85%.
- ❑ The liquid limit must be greater than 90%.
- ❑ The swelling pressure must be between 30 kg/cm² and 70 kg/cm², for a dry density of 1.60 g/cm³.

As can be observed in Table 3.3, for the tests made on the storage pile samples, the average

values for the liquid limit and the swelling pressure meet the specifications. The percentage of particles greater than 5 mm is larger than specified, but this specification is easily met by sieving the material over the 5-mm mesh. The bentonite from the storage pile was selected as it could meet the required technical specifications. Nevertheless, it was still necessary to condition the material by homogenizing, drying, and classifying the bentonite to obtain the final material to be used in the fabrication of the blocks.

3.2. Homogenization and drying in the quarry

3.2.1. Homogenization, drying and sampling

The first conditioning of the bentonite took place at the quarry site and basically consisted of homogenizing and drying, using Procedure 70-IMA-G-3-71 [33]. This conditioning is briefly described below and in detail in the reports of CIEMAT and AITEMIN:

- ❑ 70-IMA-L-3-1 *Selección, homogeneización y análisis de una partida de bentonita procedente de la cantera "La Serrata" (Almería), para el Proyecto FEBEX* [31].
- ❑ 70-AIT-L-3-01 *Proceso de homogeneización, oreado, secado y muestreo de una pila de bentonita en la mina "La Serrata" (Almería)* [39].

In the first place, a frequently used surface for the homogenization and drying of the bentonite was prepared and leveled. That base was already covered by a compacted bentonite, which facilitated the preparation of a convenient conditioning site for the operations noted below.

In the second place, the material from the storage pile was transported to the conditioning site, where it was spread in a single layer with a relatively uniform thickness of 0.30 m.

The homogenization was made using the method usually employed by Minas de Gádor:

- ❑ Plow and turn the material using tracked equipment.
- ❑ Crush the clods and homogenize using a rotary cultivator.
- ❑ Manually remove the volcanic fragments.
- ❑ Air-dry, approximately one day.

Table 3.1
Results of tests on quarry samples

Sample reference	w %	<5 mm %	<2 mm %	<74 μm %	w _L %	w _p %	I _p %	ρ _d g/cm ³	P _s kg/cm ²	SS(*) m ² / g
C-0.1	14.1	98	98	83	93			1.60	10	
C-0.2	16.9	82	98	81	81			1.59	49	
C-0.3	16.3	85	96	85	118			1.59	18	
C-0.4	21.3	100	100	96	92			1.60	77	
C-0.5	21.4	100	100	96	91			1.61	56	
C-0.6	23.0	100	100	96	95			1.57	63	
C-0.7	20.7	100	100	94	109			1.61	64	
C-1.1	16.0	100	100	97	96			1.61	56	
C-1.1.2	16.0	100	100	97	101			1.60	>62	19
C-1.3	16.0	100	100	97	102			1.58	53	19
C-1.4	14.0	95	100	91	108			1.58	>68	29
C-1.5	12.0	98	100	90	104			1.57	51	
C-A	12.0	100	100	90	88			1.61 1.60	>68 59	
C-2.1	20.0	76	99	90	101 104	52	52	1.61	43	28
C-2.2	12.0	100	99	87	99			1.60	38	32
C-2.3	17.0	99	99	93	100			1.62 1.59	>68 30	40
C-3.1	16.0	100	100	95	93			1.58	40	34
C-3.2	19.0	100	100	94	99			1.54 1.61	51 54	22
C-3.3	11.0	91	100	94	94			1.60	24	46
C-4.1	9.0	94	100	84	86			1.59	9	48
C-4.2	14.0	47	75	31	68			1.62 1.60	53 33	56
C-4.3	20.0	100	100	95	106			1.61	>57	34
C-5.2	9.0	100	100	53	65			1.59	13	51
AVERAGE	15.9	94	98	87	96	52	52	1.60	43	35
STD. DEV.	4.0	12	5	15	12			0.02	19	12

(*) SS: External specific surface, BET

Table 3.2
Results of test on samples from central test pit of the quarry

Sample reference	w %	<5 mm %	<2 mm %	<74 μm %	w _L %	ρ _d g/cm ³	P _s kg/cm ²
CC0-1	22.3	100	98	71	68	1.64	18
CC0-2	28.1	100	100	92	81	1.62	42
CC0-3	30.5	98	99	89	87	1.65	>71
CC0-4	29.4	96	97	87	78	1.60	40
CC0-5	27.4	99	98	95	96	1.69	77
AVERAGE	27.5	99	98	87	82	1.64	44
STD. DEV..	3.2	2	1	9	10	0.03	24

Table 3.3
Results of tests on storage pile samples

Sample reference	w %	<5 mm %	<2 mm %	<74 μm %	w _L %	ρ _d g/cm ³	P _s kg/cm ²
PI-1	22.9	77.0	99.5	81.5	75	1.59	48
PI-2	19.1	75.5	97.7	87.1	94	1.60	56
PI-3	21.6	88.3	97.6	86.4	92	1.56	55
PI-4	25.5	100.0	100.0	94.2	104	1.59	60
PI-5	21.3	83.5	99.4	85.6	90	1.61	48
PI-6	19.1	99.6	98.6	89.6	96	1.61	48
PI-7	26.0	98.6	99.6	80.9	80	1.60	50
PI-8	18.8	100.0	98.9	90.3	90	1.60	38
AVERAGE	21.8	90.3	98.9	87.0	90	1.60	50
STD. DEV.	2.8	10.6	0.9	4.5	9	0.02	7

The conditioning site can be seen in Figures 3.2 and 3.3 as it was during some of these operations.

In each cycle of conditioning (homogenizing and drying), the size of the clods was reduced. After four cycles, it was considered that the required result had been obtained.

After these four cycles of conditioning, samples were taken to check, by laboratory tests, the characteristics and homogeneity of the conditioned bentonite. To obtain these samples, the edges were ignored and an area of 1200 m² with an average thickness of 0.30 m was delineated with stakes. This area was then divided into 48 squares

of 25 m² each, in accordance with Procedure 70-IMA-G-3-71 [33], and in each square a sample of 3 to 4 kg was taken. The samples, designated P-1 to P-48, were packaged in plastic bags to be sent to the CIEMAT laboratory.

Once the samples were taken, the material was gathered into a pile to await the results of the laboratory tests. If the results indicated that the homogenization met the requirements, the bentonite could then be transported to the factory for further conditioning; if not, then more cycles would take place at the conditioning site.

3.2.2. Laboratory tests

One-half of each of the 48 samples, P-1 to P-48, was taken and mixed together to form a new sample, which was then successively quartered to obtain 10 aliquots, designated H-1 to H-10. From these 10 and another 11 unmixed, from the remaining halves of the 48 samples, laboratory tests were made: water contents, grain-size determinations, Atterberg limits, and swelling pressures. The results are shown in Table 3.4.

As can be seen in the table, the average value of each property is very similar in the two sets of sam-

ples (H and P) and the values also meet the specifications. The standard deviation is less for the mixed sample; therefore, it appears that further homogenization took place in the laboratory mixing. It was considered, however, that for all practical purposes, for a semi-industrial scale, the degree of homogenization achieved in the field was adequate, thus the bentonite was sent to the planned factory conditioning.

3.3. Factory conditioning, bagging, and sampling

Around 600 tons of bentonite were taken to the factory for conditioning. The conditioning was made using Procedure 70-IMA-G-3-71 [33]. The various operations and controls are described in the following reports:

- ❑ 70-IMA-L-3-10 *Proceso de una partida de bentonita en Fábrica de Minas de Gádor (Almería) para el Proyecto FEBEX* [40].
- ❑ 70-AIT-L-3-02 *Procesado de bentonita en la planta de Minas de Gádor* [41].



Figure 3.2: View of the conditioning site on the second day of the homogenizing process.



Figure 3.3: View of the conditioning site after a pass of the rotary cultivator.

The operations included in the factory conditioning were: crushing, drying, classification, bagging, and sampling.

Before starting these operations, the factory was adapted for the process and a test was made to establish the optimum conditioning regime.

3.3.1. Crushing

A bladed crusher was used to reduce the material to a maximum size of 10 cm. The crusher was fed by a conveyor belt from the bentonite that had been piled up nearby.

After the material had been crushed, it was transported by a lineal feed to equipment, employing both smooth rollers and bladed rollers, to obtain a granulated material with a maximum size of 5 cm.

The procedure established that if, after crushing, the water content was found to be between 13% and 15%, the material could be sent directly to the classification operation, but as the average value of the water content was found to be 21%, the bentonite was sent to the drying process.

3.3.2. Drying

The material was transported by an adjustable conveyor belt to a 20 m long, 1.5 m in diameter, rotary dryer. The clay proceeded through the dryer by the actions of revolving blades, gravity, and rotation.

The dryer was kept at a temperature between 50°C and 60°C so that, after drying and in accordance with the specifications, the water content was $13 \pm 2 \%$, as shown by systematic control tests.

Also control tests were made to determine the absorption of humidity by the bentonite during the time between the drying cycle and the bagging process. The results obtained indicated that the water content increased two percent for each 12 hours of exposure to the air.

The bentonite was raised by means of a bucket elevator from the rotary dryer to a storage silo.

3.3.3. Classification

The system of classification consisted of two parts: feeding and sieving. The feeding was by

Tabla 3.4
Results of tests on storage pile samples, after homogenization

Sample reference	w %	<5 mm %	<2 mm %	<74 μm %	w _L %	w _P %	I _P %	ρ _d g/cm ³	P _s kg/cm ²	SS(*) m ² /g
H-1	22.2	94.1	98.5	85.6	99	50	49	1.63	54	
H-2	22.5	95.3	98.1	86.1	98	50	48	1.60	44	
H-3	22.1	98.8	97.0	85.2	97	51	46	1.60	48	
H-4	21.8	98.3	98.2	89.2	96	47	48	1.59	>58	
H-5	21.8	98.7	97.8	85.8	101	50	51	1.59	45	
H-6	22.2	94.2	98.9	87.5	101	50	51	1.60	47	
H-7	21.7	97.9	98.6	86.9	96	49	47	1.60	57	
H-8	21.6	97.4	97.5	88.9	103	50	53	1.61	32	
H-9	21.5	98.9	99.0	85.6	97	51	46	1.61	36	
H-10	21.4	98.6	98.6	88.1	95	48	47	1.62	>54	
AVERAGE	21.9	97.2	98.2	86.9	98	50	49	1.61	45	
STD. DEV.	0.4	1.9	0.6	1.5	3	1	2	0.01	8	
P-1	21.7	98.4	98.8	88.3	93	49	44	1.61	39	33
P-10	20.5	92.7	96.6	87.5	104	52	52	1.60	34	37
P-16	20.3	98.4	98.7	87.0	97	50	47	1.59	59	38
P-18	22.4	96.3	97.4	85.5	104	54	50	1.60	46	34
P-24	19.5	100.0	97.3	86.6	98	56	42	1.60	40	40
P-26	21.8	100.0	97.1	84.3	100	56	44	1.61	60	40
P-31	25.5	96.9	98.8	80.3	93	51	42	1.60	40	39
P-38	25.9	99.5	97.1	81.3	89	48	41	1.59	52	35
P-40	24.8	98.6	98.1	85.0	91	51	40	1.61	>54	38
P-47	19.8	96.8	98.1	84.0	97	49	47	1.62	>67	41
P-7	23.4	97.7	97.2	84.1	100	50	50	1.63	>62	39
AVERAGE	22.3	97.8	97.7	84.9	97	51	45	1.61	46	38
STD. DEV.	2.3	2.1	0.8	2.5	5	3	4	0.01	10	3

(*) SS: External specific surface, BET

means of a bucket elevator and a hopper with an adjustable trapdoor; it was necessary to maintain a constant volume of material arriving at the sieve to avoid some fines being dragged away with the rejected material. The sieving was done over a 5-mm mesh with a controlled inclination and the final material (that less than 5 mm in size) fell, by gravity, to the bagging bin.

3.3.4. Bagging and sampling

The system of bagging consisted of a receiving hopper from which the impermeable big-bags, each with a capacity of 1300 kg, were filled. Then the big-bags were removed by a lifting truck and taken to their place of storage. A total of 233 big-bags were filled, with an average weight of 1280 kg; or 298 tons of bentonite resulted from

processing in accordance with the specifications established.

One of the conditions that was established for quality control was that the material must not be exposed to air at ambient temperature in the silo for more than four hours, in order to prevent the absorption of humidity as indicated in Section 3.3.2. Another condition established was that the water content of the material at the moment of bagging should be between 12.5% and 15.5%. The water content was determined in 27% of the big-bags on samples of 0.5 kg taken during the bagging process.

In addition, during the bagging process, a sample of 8 to 10 kg was taken approximately every 2.5 tons; in total, 119 samples designated 70-IMA-3-4-1 to 70-IMA-3-4-119 were taken. These samples were to be used both for the detailed characterization of the bentonite and for the set of experimental laboratory tests.

4

CHARACTERIZATION TESTS

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As stated in Section 3.3.4, after conditioning the mass of bentonite, samples were taken of the final material to be used in making the complete characterization tests on the material to be employed in the fabrication of the blocks and in performing the set of experimental laboratory tests.

The characterization tests referred to herein were made by CIEMAT, UPC-DIT, and CSIC-Zaidín and the results have been presented in these reports:

- ❑ CIEMAT: 70-IMA-L-3-15 *Characterisation of "La Serrata" clay used in the FEBEX Project* [42].
- ❑ UPC-DIT: 70-UPC-L-3-01 *THM laboratory tests in FEBEX Phase 3* [43]
- ❑ CSIC-Zaidín: 70-ZAI-L-03-02 *Caracterización mineralógica y geoquímica de la bentonita inicial. Proyecto FEBEX* [44]

4.1. Samples for laboratory tests

The samples taken during the bagging of the final material were assigned the references 70-IMA-3-4-1 to 70-IMA-3-4-119, packaged in plastic bags, and sent to the laboratory of CIEMAT. One-half of each of these samples was mixed to form a homogenized sample designated 70-IMA-3-4-0.

The homogenized sample was the one from which the material was sent to the laboratories of the two other participating institutions (UPC-DIT and CSIC-Zaidín).

In the CIEMAT laboratory, tests were made on various of the unmixed samples (70-IMA-3-4-1 to 70-IMA-3-4-119) to check the variability of the parameters. To simplify these references, the designation F- α is used for the test samples, where α is the number of the sample 70-IMA-3-4- α from which the tested sample came; for example, sample F-17 is really sample 70-IMA-3-4-17. In addition, tests were made on five aliquots, called A-1 to A-5, of the homogenized sample 70-IMA-3-4-0, to compare both with the results of the F samples and with similar tests made by the other laboratories. In the tests made by the CIEMAT laboratory, therefore, the tests on samples that were unmixed (F samples) and the one that was homogenized (A samples) are differentiated, whereas the other two

laboratories utilized only parts of the homogenized sample.

4.2. Identification properties

The data that are presented in this section are being called the identification properties (following the geotechnical terminology) because they give an idea, by means of simple tests, of the type of physico-chemical behavior of a clay.

4.2.1. Water content

The water contents were determined on samples which continued to be kept in closed plastic bags. From the results, presented in Table 4.1, it can be observed that the average water content in the unmixed samples (F samples) is slightly less than that determined in the aliquots of the homogenized sample (A samples), i.e. 13.4% and 14.5%, respectively.

4.2.2. Atterberg limits

The Atterberg limits were determined on 14 of the unmixed samples (F samples) and on 5 (A-1 to A-5) of the homogenized sample. This determination was made using Procedure 70-IMA-G-0-12 [36]. The results are also presented in Table 4.1.

In addition, the Atterberg limits were determined by UPC-DIT, following the standards of the Laboratorio del Transporte y Mecánica del Suelo (CEDEX NLT/105/72 [45] and NLT 106/72 [46]). The results are presented in Table 4.2.

The average value of the liquid limit obtained by UPC-DIT is somewhat less (5 %) than that obtained by CIEMAT in the homogenized sample; however, the determinations of the liquid limit for a clay can change with variations in the standard testing methods used for clays, especially for one as plastic as bentonite. UPC-DIT made only 5 determinations, whereas CIEMAT has had a lot of experience in determining the Atterberg limits for bentonite from this deposit and, therefore, it has been considered that the results obtained by CIEMAT are more representative of the material.

In any case, a liquid limit on the order of 100 % seems very low for a bentonite with a content of montmorillonite greater than 90 % (as shown in Section 4.3) and with a large proportion of sodium as an exchangeable cation.

Table 4.1
 Identification properties

Sample reference	w %	<74 μm %	<2 μm %	w _L %	w _P %	I _P	Specific weight	SS (*) m ² /g
F-2	14.8	93	65	103	52	49	2.75	28
F-4	15.1			105	53	48		31
F-17	16.0	92	69	100	53	46		34
F-18	15.0	92	67	111	53	48	2.62	38
F-22	15.1			110	52	51		35
F-37	13.3	93	69	100	52	51	2.67	35
F-58	11.9	92	59	101	54	47	2.75	30
F-59	12.7	92	66					31
F-65	11.9			108	51	47	2.77	26
F-75	11.7	92	63	96	51	45	2.68	32
F-79	12.3			101	50	51		33
F-93	13.1	93	64	104	47	57	2.73	26
F-102	14.1	93	66	103	52	51	2.65	33
F-109	11.9	93	66	100	47	53	2.72	34
F-119	12.3	93	67	103	51	52	2.70	35
AVERAGE	13.4	93	65	103	51	50	2.70	32
STD. DEV.	1.5	1	3	4	2	3	0.05	3
A-1	14.4			98	54	44		28
A-2	14.7			98	55	52	2.68	30
A-3	14.4			98	55	47	2.75	32
A-4	14.3			100	60	50	2.72	32
A-5	14.5			98	58	42		29
AVERAGE	14.5			98	56	47	2.72	30
STD. DEV.	0.2			1	3	4	0.04	2

(*) SS: External specific surface, BET.

Table 4.2
Atterberg limits

Sample	Liquid limit	Plastic limit	Plasticity index
n-1 (*)	94.5	46.4	48.1
n-2	92.5	46.8	45.7
n-3	92.0	48.4	43.6
n-4	91.5	45.3	46.2
n-5	94.2	46.3	47.9
AVERAGE	92.94	46.64	46.30
STD. DEV.	1.34	1.13	1.83

(*) Name given by UPC-DIT to tested parts of sample 70-IMA-3-4-0 received from CIEMAT

4.2.3. Grain-size distribution

Grain-size determinations were made on 11 unmixed samples (F samples): by sieving for the particle fraction greater than 74 μm , following Procedure 70-IMA-G-0-05 [35], and by sedimentation for the particle fraction less than 74 μm , following Procedure 70-IMA-G-0-06 [47]. Prior to the grain-size determinations, the samples were hydrated with distilled water and a sodic hexameta-phosphate solution for 18 hours and the samples were subjected to alternating cycles of stirring and ultrasound to aid dispersion.

Figure 4.1 shows the average grain-size curve with an indication of the standard deviations and Table 4.1 shows both the fractional content of particles less than 74 μm and less than 2 μm . The grain-size determinations indicate that the fraction less than 2 μm (clay size) averages about 65%, that 28 % is silt size (particle sizes between 2 μm and 74 μm), and that the remaining 7 % is sand size (greater than 74 μm).

The laboratory of UPC-DIT also made grain-size determinations on 5 samples (n-1 to n-5, aliquots of the 70-IMA-3-4-0 sample received from CIEMAT), by sieving for the particles larger than 74 μm , following the Standard NLT 104/72 [48], and by means of sedimentation for the particles less than 74 μm , following the British Standards Procedure 1377:1975 [49].

The curves obtained for the five samples are represented in Figure 4.2, where it can be observed that the percentage averages about 45 for the grain sizes less than 2 μm (clay size), 42 % for the silt size (between 2 μm and 74 μm), and 13 % for the sand size.

The results from the two laboratories are very different, but these differences are due to the form of preparation of the sample, prior to making the determinations, since the results of grain-size determinations of a clay of high plasticity are very dependent on the degree of dispersion prior to the test. Nevertheless, the sensitivity of the results of grain-size determinations and of the Atterberg limits (especially the liquid limit) to the treatment of the sample is an indicator of the physical behavior of the bentonite. The behavior can vary for samples from the same deposit, with the same mineralogic composition, but in a different state of aggregation.

4.2.4. External specific surface

These tests were performed on 15 unmixed samples (F samples) and 5 from the homogenized sample (A samples), following Procedure 70-IMA-G-0-11 [38]. In this method, nitrogen is used as the absorbent gas as it does not occupy the inter-layer space and thus the result is referred to only as the external specific surface. The calculation was made by BET method of the single point.

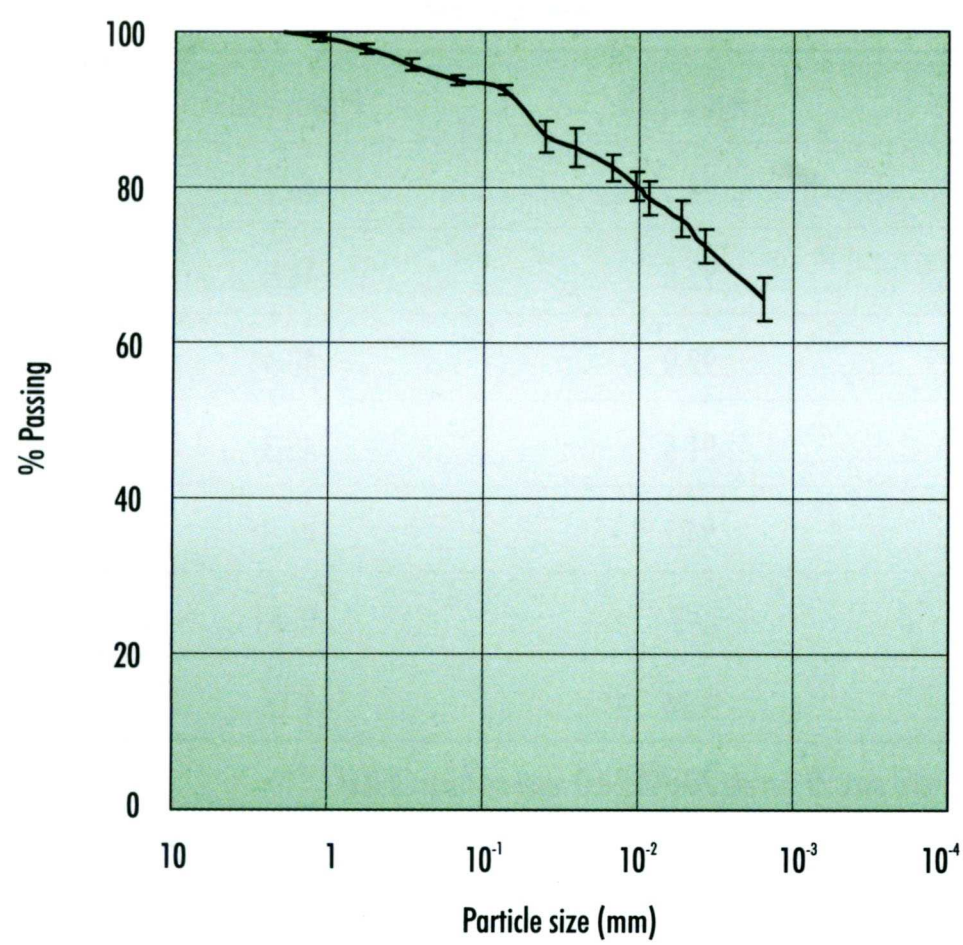


Figure 4.1: Average grain-size curve of the 11 samples analyzed, with indication of standard deviations.

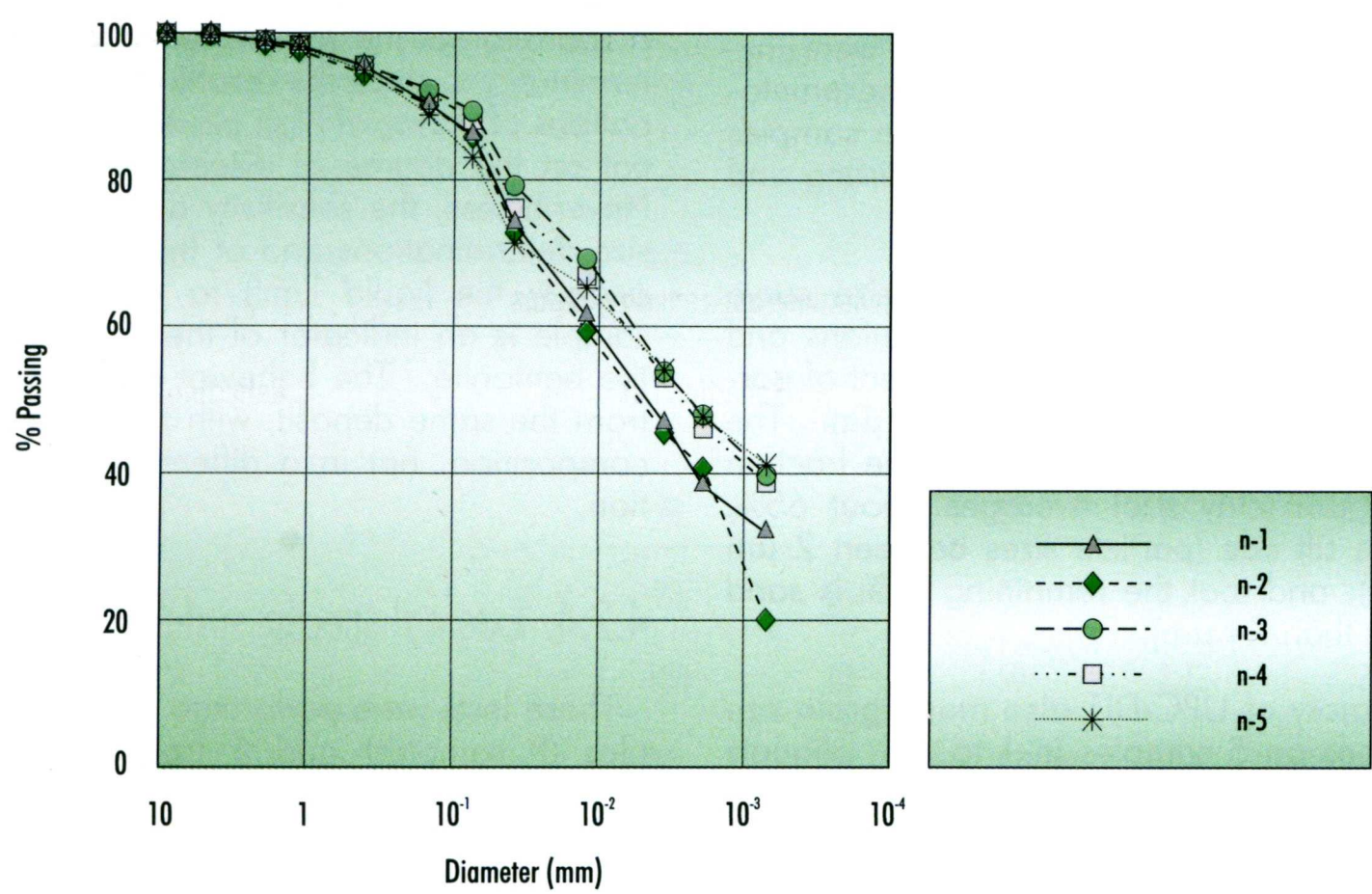


Figure 4.2: Grain-size distribution curves.

The results are shown in Table 4.1 with average values of the external specific surface (BET) of 32 m²/g and 30 m²/g for the F and A samples, respectively. These values of the external specific surface are very low for a bentonite with a montmorillonite content greater than 90% (Section 4.3.).

4.2.5. Specific weight

The specific weight was determined for 10 un-mixed samples (F samples) and 3 from the homogenized sample (A samples). Bi-distilled water, as the means of suspension, and classic picnometers were used for this determination, following Procedure 70-IMA-G-0-9 [50].

The results are presented in Table 4.1 with average values of 2.70 and 2.72 for the F and A samples, respectively. The specific weight obtained was less than the average value from the analyses made in the years prior to FEBEX for the bentonite from the same deposit (Chapter 2).

4.3. Mineralogical and chemical characterization

The mineralogical characterization was made on ten of the un-mixed samples (F samples) and on 5 from the homogenized sample (A samples) by the CIEMAT laboratory and three aliquots of the homogenized sample by the CSIC-Zaidín laboratory.

The chemical analyses were performed on four type F and two type A samples by the CIEMAT laboratory and four aliquots of the homogenized sample by the CSIC-Zaidín laboratory.

4.3.1. Mineralogical characterization

4.3.1.1. X-ray diffraction

Methods of analysis

For their preparation, the samples were dried at 60°C and then ground to obtain a grain size less than 60 μm.

The content of the essential minerals was determined in a bulk sample by X-ray diffraction (XRD), with a PW-1370 Philips diffractometer and the powder method on a flat-rotating sample holder. The exploration velocity was 2°/min with 2θ in the range between 2° and 70°. The semiquantification was made using the reflecting factors, taken from Schultz (1964) [51] and Barahona (1974) [52], that are shown in Table 4.3.

After being identified and separated, using a stereoscopic microscope, the accessory minerals were determined by using a scanning electronic microscope (SEM) coupled to an energy dispersive X-ray analysis system.

The fraction of less than 2 μm was obtained by suspension, sedimentation, and filtration. Then the

Table 4.3
Reflections and reflecting factors

Mineral	Reflection (Å)	Reflecting factor
Total phyllosilicates (smectite,Sm)	4.45	0.1-0.15
Quartz (Qz)	3.34	1.5
K-feldspar (K-Fd)	3.24	1.0
Plagioclase (Plag)	3.18	1.0
Cristobalite (Cri)	4.07	0.6
Tridimite (Tri)	4.30	0.6
Calcite (Cc)	3.03	1.0

oriented clay mineral aggregates (OA) were prepared, treated with ethylene glycol and sulfoxide dimethyl, and heated to 300°C and 550°C to determine the clay minerals in the less than 2 μm fraction by XRD.

In addition, analytical determinations were made by CSIC-Zaidín, using similar methods. The samples were dried at 60°C, then ground to pass the ASTM 270-mesh sieve (53 μm).

The mineralogic analysis was also made with a PW-1730 Philips diffractometer, with graphite monochromator and exploration velocity of 2°/min. A Philips 1710/100 diffractometer with radiation Cu-K α , graphite monochromator, and an exploration velocity of 1.2°/min was also utilized.

The study of the bulk sample was made on a flat-rotating sample holder and powder patterns were obtained from 2° to 80° of 2 θ . For the semiquantitative calculation of the minerals present in the samples, the areas of the diagnostic peaks of the minerals were measured over the powder pattern and corrected by the reflecting factors. The values of the reflecting factors are indicated above in Table 4.3. For the fraction less than 2 μm , separated using sedimentation, the technique of oriented aggregates (OA) by sedimentation at ambient temperature was used and the OA treated with ethylene glycol (OA + EG). In the latter case, the sample was balanced with ethylene glycol vapor at 60°C in an appropriate desiccator.

Bulk sample — Results

The semiquantitative mineralogical composition of the bulk sample is shown in Table 4.4. The results show that the principal mineralogical association is formed by smectite (Sm), quartz (Qz), plagioclase (Plag), and cristobalite (Cri). The potassium feldspar (K-Fd), the tridymite (Tri), and the calcite (Cc) usually appear as traces.

Smectite is the principal mineral and its content averages 93% for the F samples (unmixed) and 92% for the A samples (homogenized).

The XRD patterns of the different samples are represented in Figures 4.3 to 4.7.

Among the accessory minerals detected by the scanning electronic microscope were: carbonates (dolomite, calcite, barite, celestine), phosphates (apatite, xenotime; monazite), oxides (ilmenite, magnetite), micas (sericite, biotite, chloritized bio-

tite), silicates (zircon, plagioclase, Al-silicates with Fe and Mg, quartz and opal), and sulfurs (pyrite).

The powder patterns obtained in the laboratory of CSIC-Zaidín are represented in Figures 4.8, 4.9, and 4.10. The smectite content is 94% and, according to reflection (060), the smectite is dioctahedric.

The estimations of the smectite content from the two laboratories are remarkably similar and both show that it is extraordinarily high.

Fraction less than 2 μm — Results

The mineralogic composition of the fraction less than 2 μm indicated that the samples were almost pure smectite: the peak observed in the untreated oriented aggregates (OA) at 14.5 Å shifted to a very strong reflection, at approximately 16.9 Å, when treated with ethylene glycol and collapsed to 10 Å when heated to 300°C.

The XRD patterns of both the treated and untreated OA samples, together with those obtained by CIEMAT from the Universidad Autonoma de Madrid (UAM), for comparative purposes, are shown in Figures 4.11 through 4.16.

The patterns of unsolvated OA samples, and solvated with ethylene glycol OA+EG, from the tests made in the laboratory of CSIC-Zaidín, are represented in Figures 4.17 through 4.22.

The patterns of OA samples solvated with ethylene glycol, as can be observed in Figures 4.18, 4.20, and 4.22, show the presence of between 10% and 20% of interstratified smectite/illite (using the method of Moore and Reynolds [53]) based on the distance between peaks 001/002 and 002/003; thus, it can be concluded that the smectite being utilized is dioctahedral and contains around 15% of interstratified illite/smectite.

4.3.1.2. Differential thermal analysis (DTA) and thermogravimetric analysis (TGA)

The thermal characteristics of the bulk sample have been determined in samples A-1 and A-5 (homogenized sample), using a Perkin-Elmer 1700 DTA apparatus at a scan rate of 10°C/min, under a dynamic air atmosphere (40 cm³/min) at temperatures between 40°C and 980°C.

Table 4.4
Semiquantitative mineralogical composition of bulk sample

Sample reference	Sm %	Qz %	Plag %	Cri %	K-Fd %	Tri %	Cc %
F-2	89	2	2	2	0	1	1
F-18	92	2	3	3	0	Tr(*)	Tr
F-37	92	3	2	2	Tr	Tr	Tr
F-58	96	2	1	1	Tr	Tr	Tr
F-65	94	2	2	2	0	Tr	Tr
F-75	89	3	4	2	0	Tr	2
F-93	95	1	2	2	Tr	2	Tr
F-102	95	2	2	1	0	Tr	Tr
F-109	93	1	3	2	0	Tr	Tr
F-119	92	2	2	3	1	Tr	Tr
AVERAGE	93±2	2±1	3±1	2	Tr	Tr	Tr
A-1	81	3	10	Tr	3	Tr	2
A-2	94	1	1	2	0	1	Tr
A-3	95	1	1	3	0	Tr	1
A-4	92	3	2	2	1	Tr	Tr
A-5	96	2	2	3	0	Tr	Tr

(*) Tr = traces

The DTA curves show an important endothermic peak at 130°C followed by an accessory peak at 200°C, corresponding to the loss of moisture. A second endothermic peak at 640°C corresponds to a loss of structural OH⁻ of the montmorillonite and another peak at 860°C can be interpreted as structural changes in the clay.

These curves, which are shown in Figures 4.23 and 4.24, correspond to a dioctahedral smectite of the montmorillonite type (MacKenzie, 1970) [54].

DTA tests were also made in the laboratory of CSIC-Zaidín, simultaneously with the thermogravimetric analysis (TGA). Both patterns are represented together in Figure 4.25.

This technique permits the detection of the reactions that are produced during the heating of the sample and the corresponding changes in weight. The analysis was made in a NETZSCH STA 409 EP apparatus, in air atmosphere, and in alumina crucibles, using alumina as a reference.

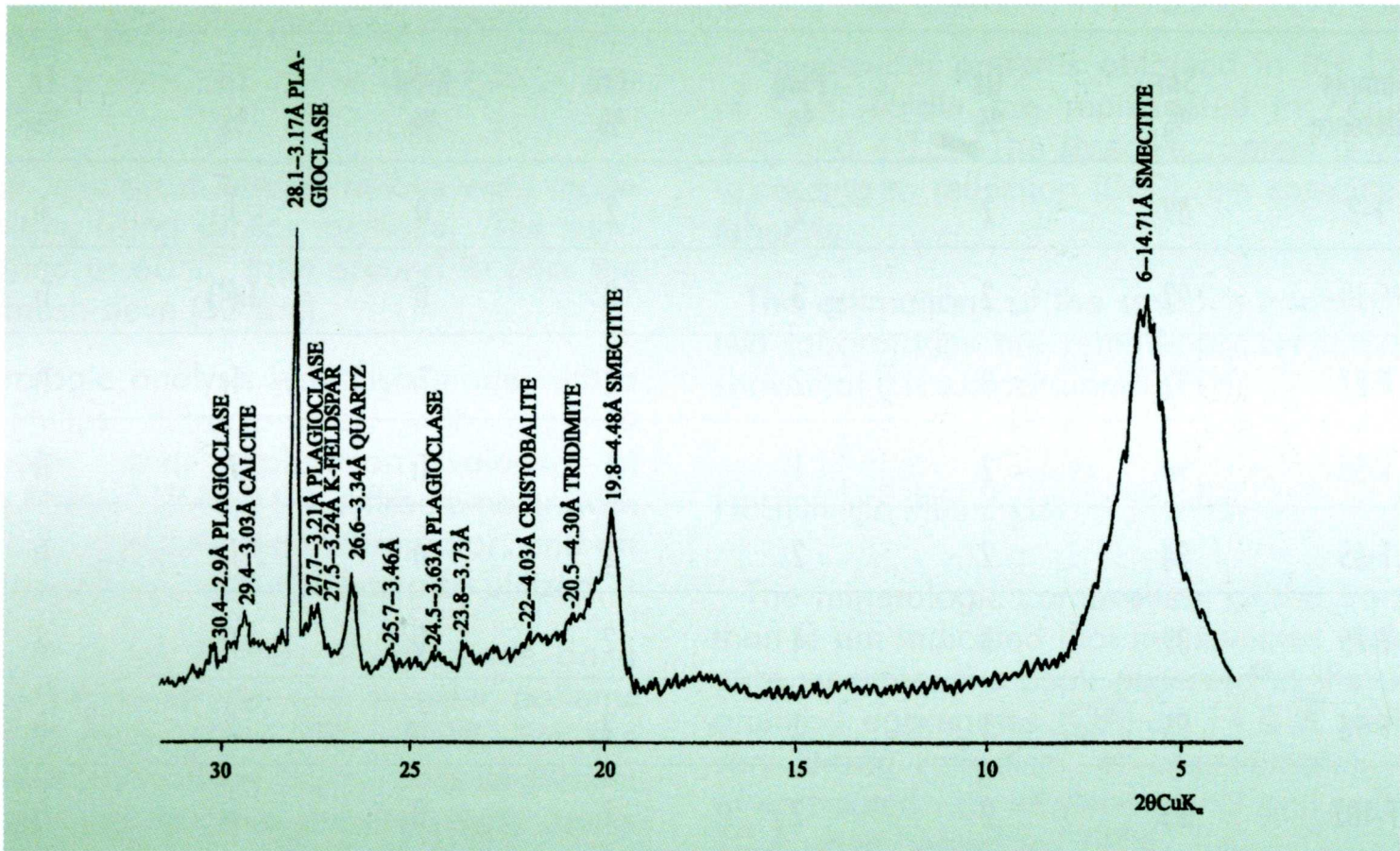


Figure 4.3: X-ray diffraction pattern (XRD) of the sample A-1 (bulk sample).

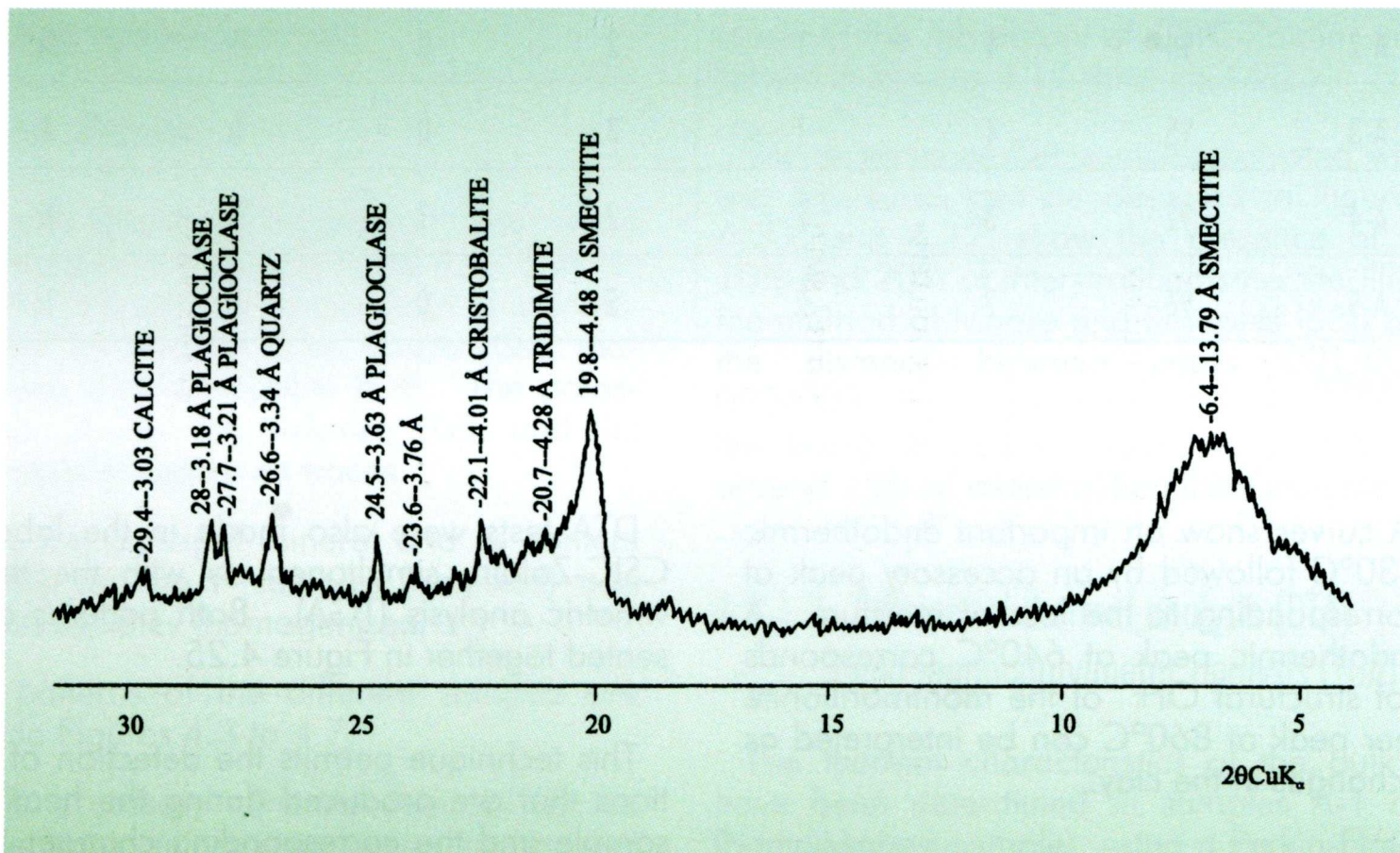


Figure 4.4: X-ray diffraction pattern (XRD) of the sample A-5 (bulk sample).

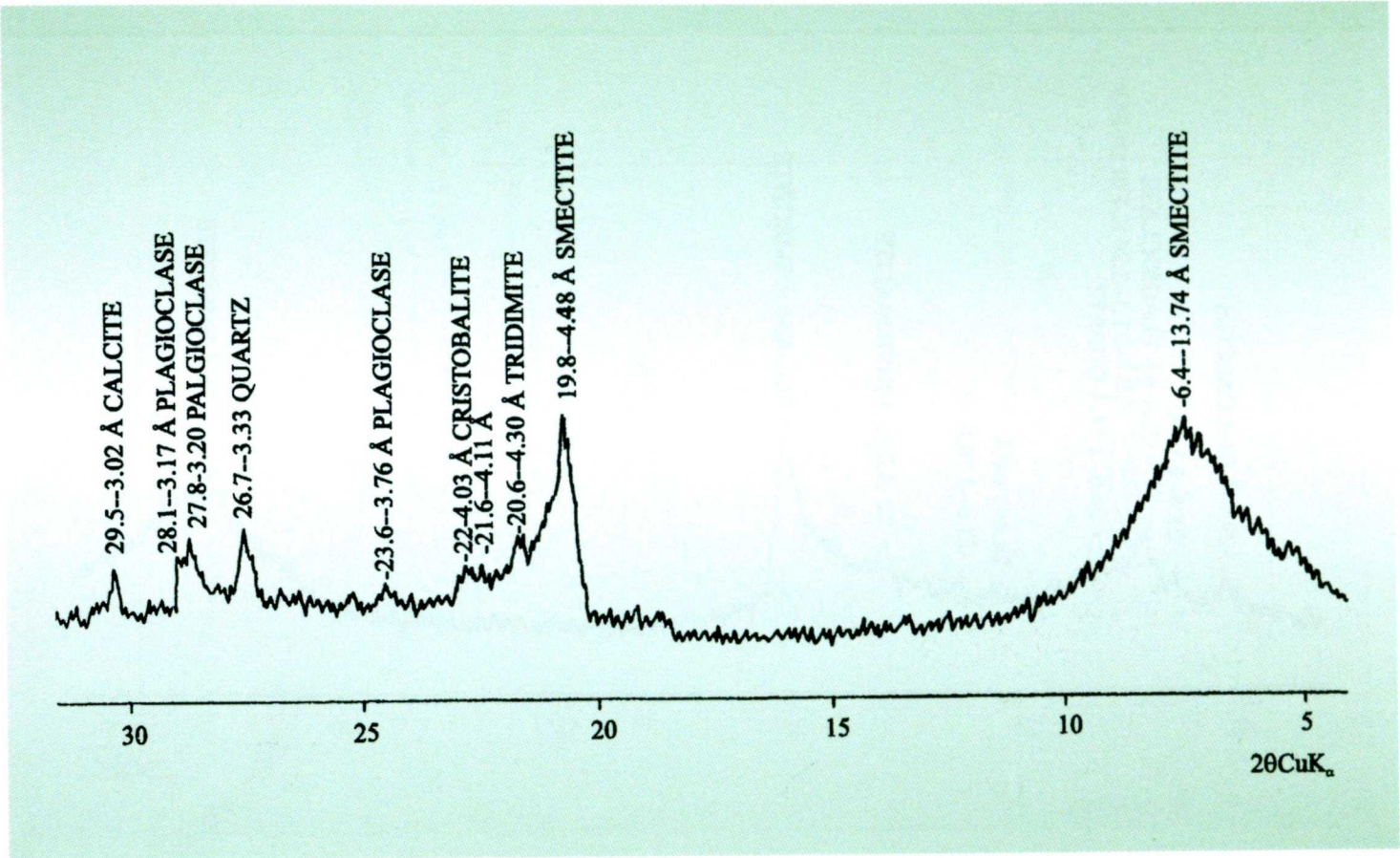


Figure 4.5: X-ray diffraction pattern (XRD) of the sample F-2 (bulk sample).

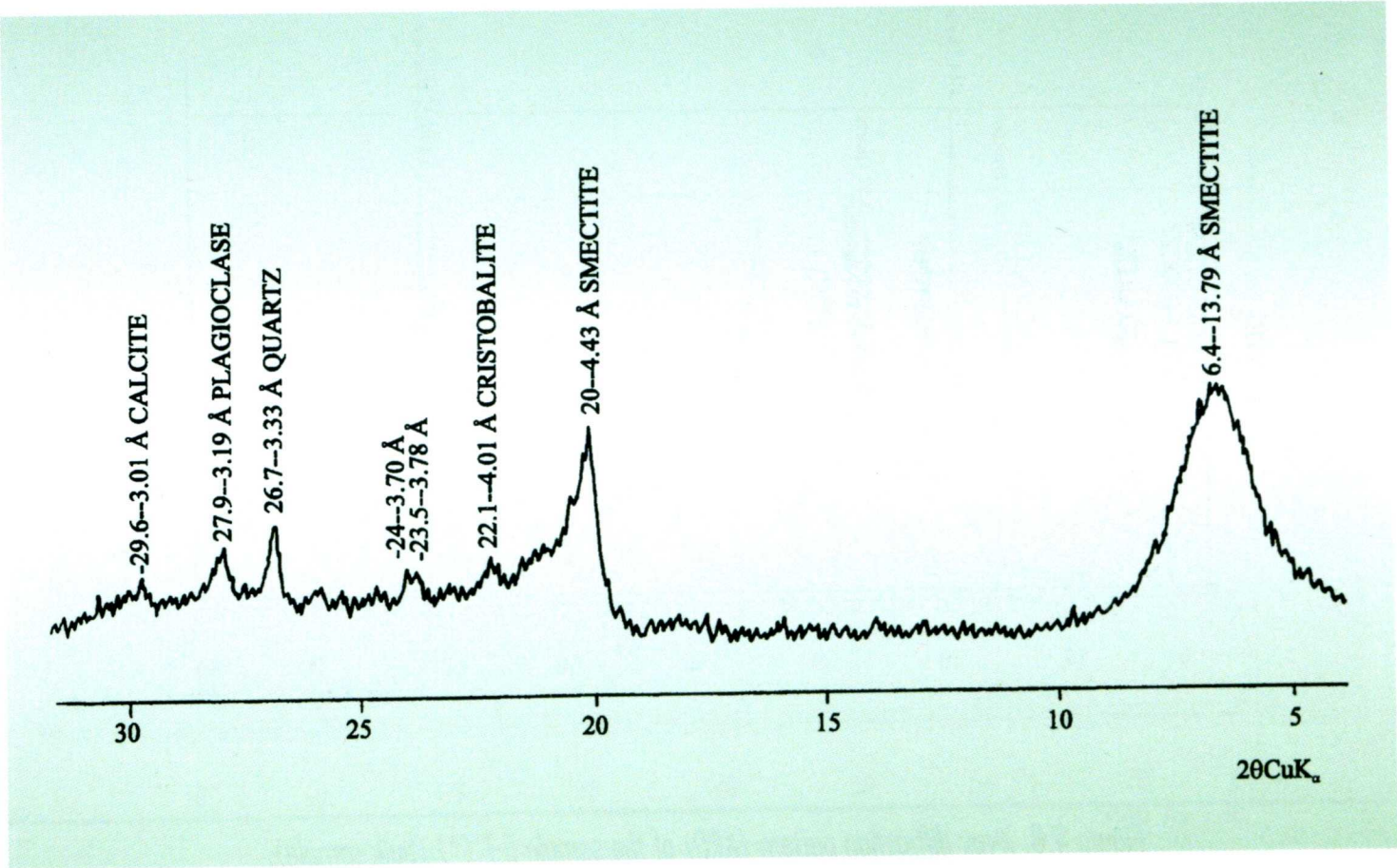


Figure 4.6: X-ray diffraction pattern (XRD) of the sample F-65 (bulk sample).

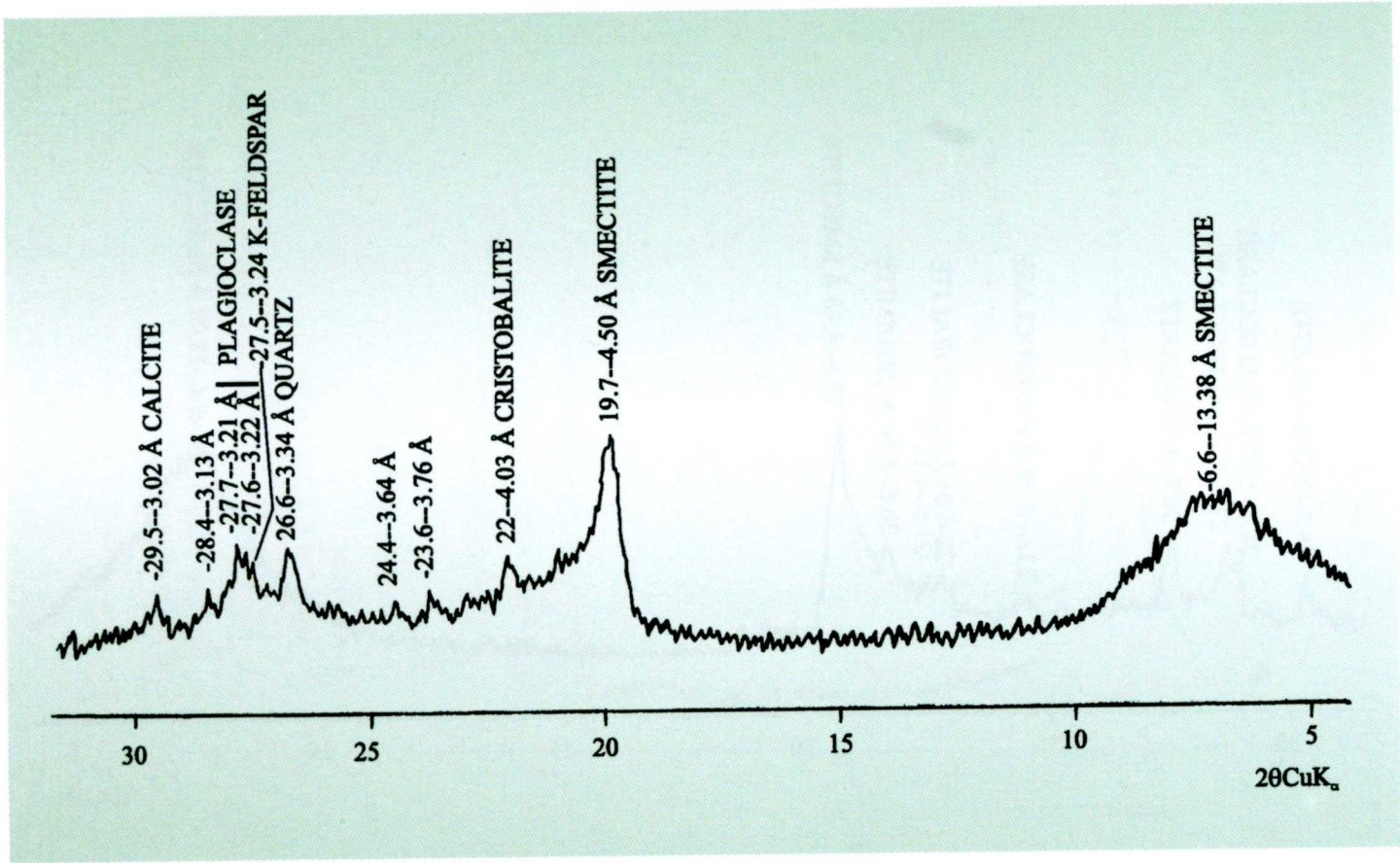


Figure 4.7: X-ray diffraction pattern (XRD) of the sample F-119 (bulk sample).

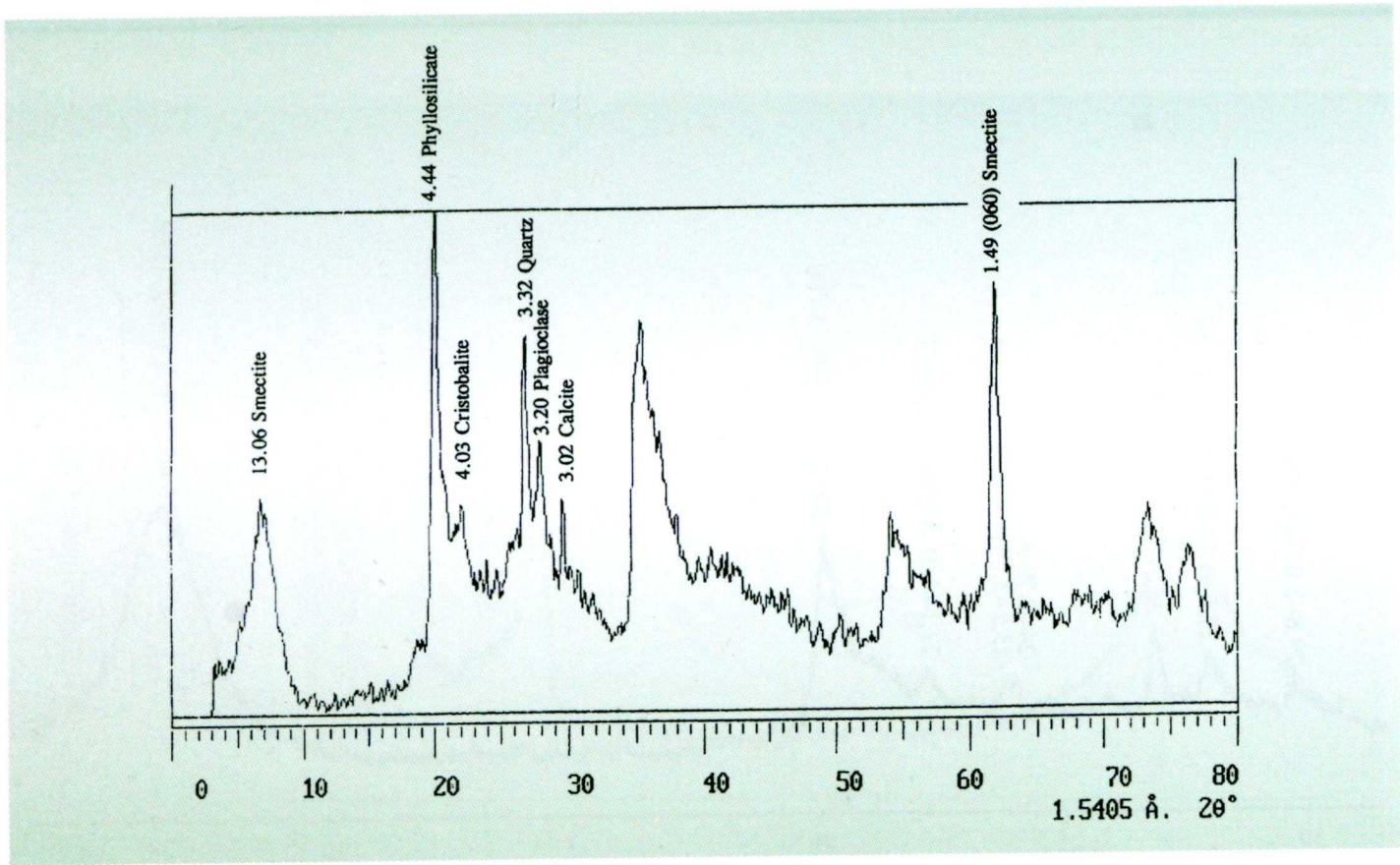


Figure 4.8: X-ray diffraction pattern (XRD) of the sample E-1 (*) (bulk sample).

* E-1, E-2 and E3 are names given by CSIC-Zaidín to the tested samples, that are aliquots of sample 70-IMA-3-4-0 received from CIEMAT.

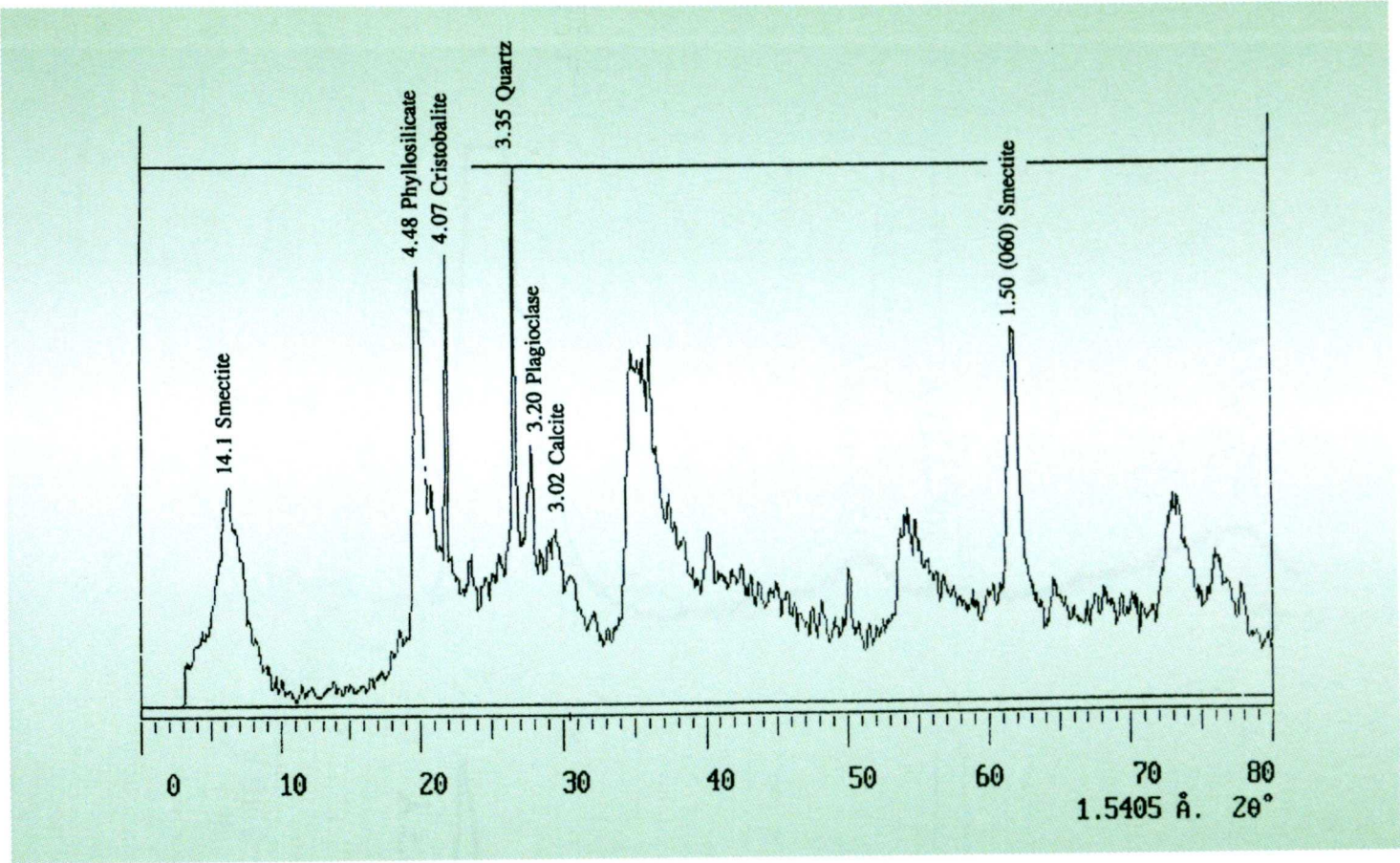


Figure 4.9: X-ray diffraction pattern (XRD) of the sample E-2 (bulk sample).

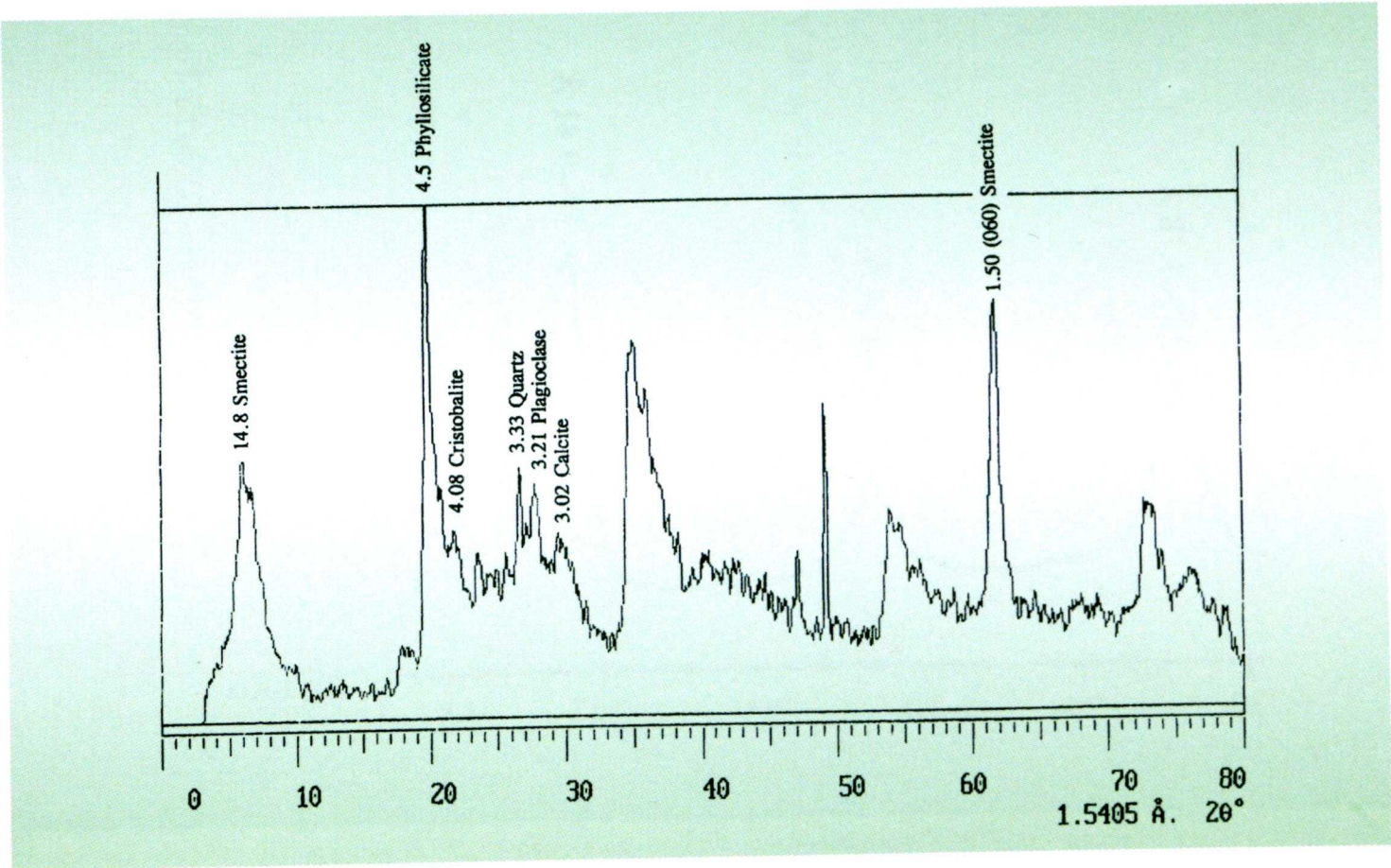


Figure 4.10: X-ray diffraction pattern (XRD) of the sample E-3 (bulk sample).

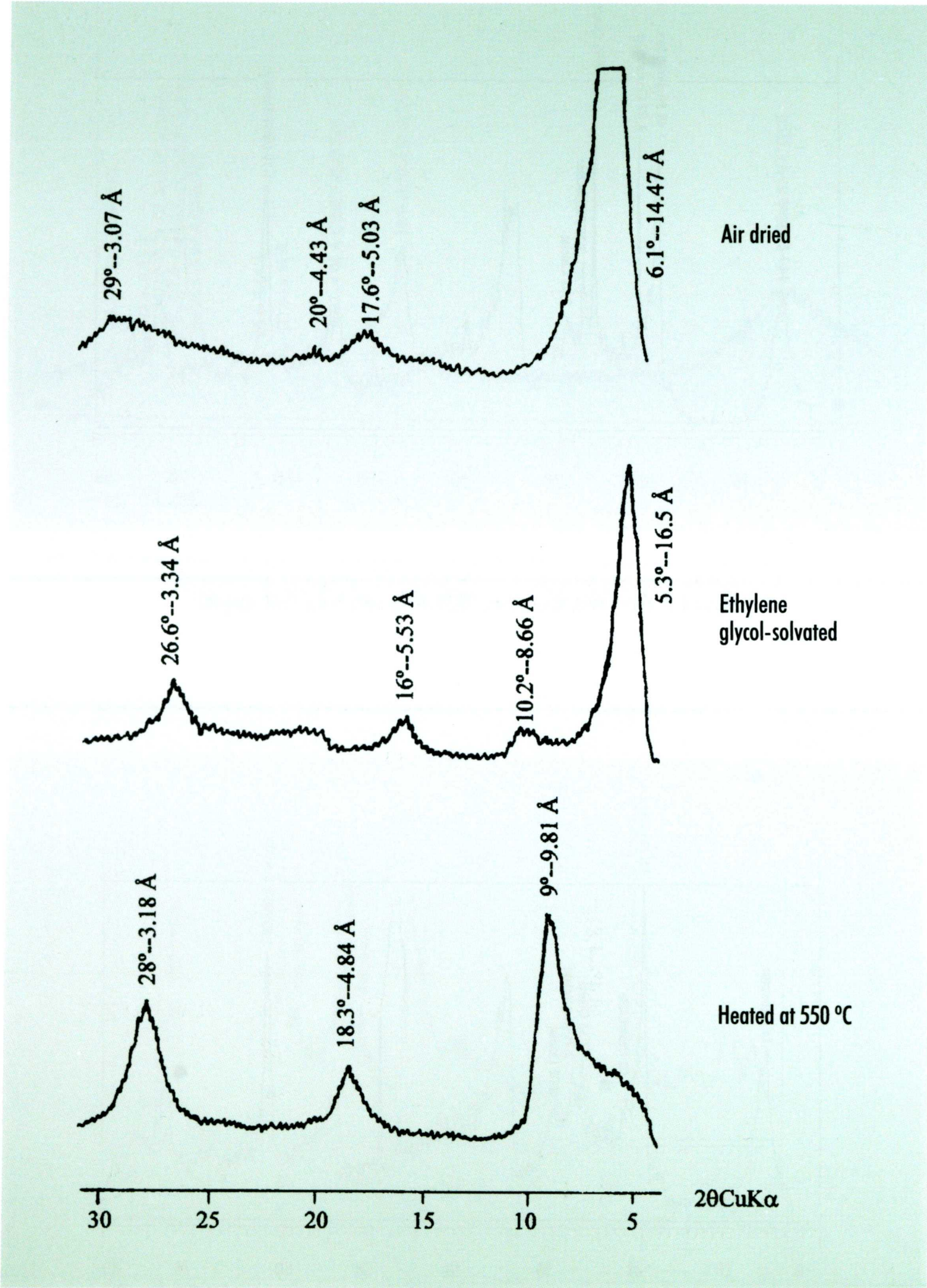


Figure 4.11: X-ray diffraction pattern (XRD) of the sample A-1 (oriented aggregate of the fraction less than 2 μm).

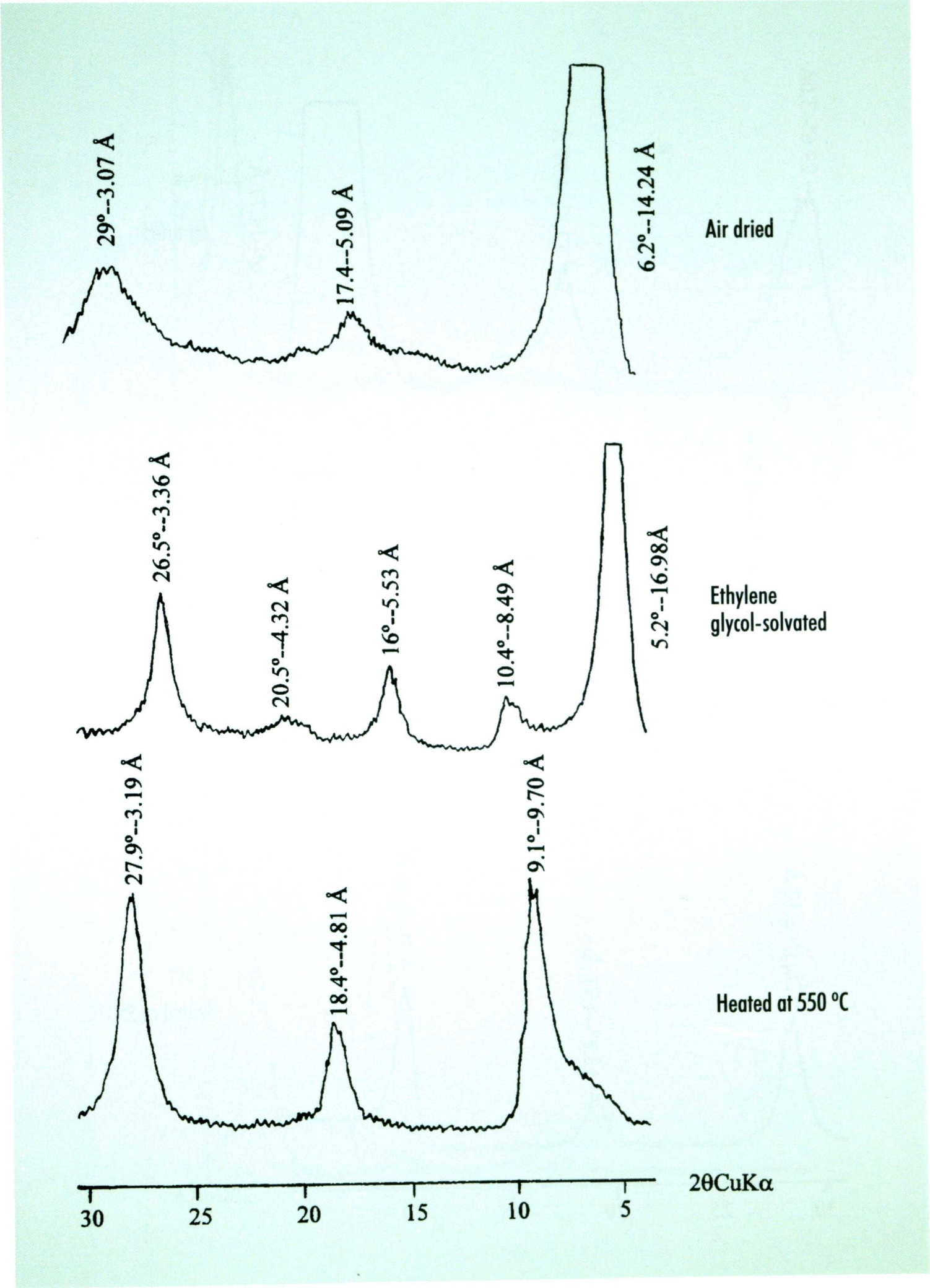


Figure 4.12: X-ray diffraction pattern (XRD) of the sample F-2 (oriented aggregate of the fraction less than 2 μm).

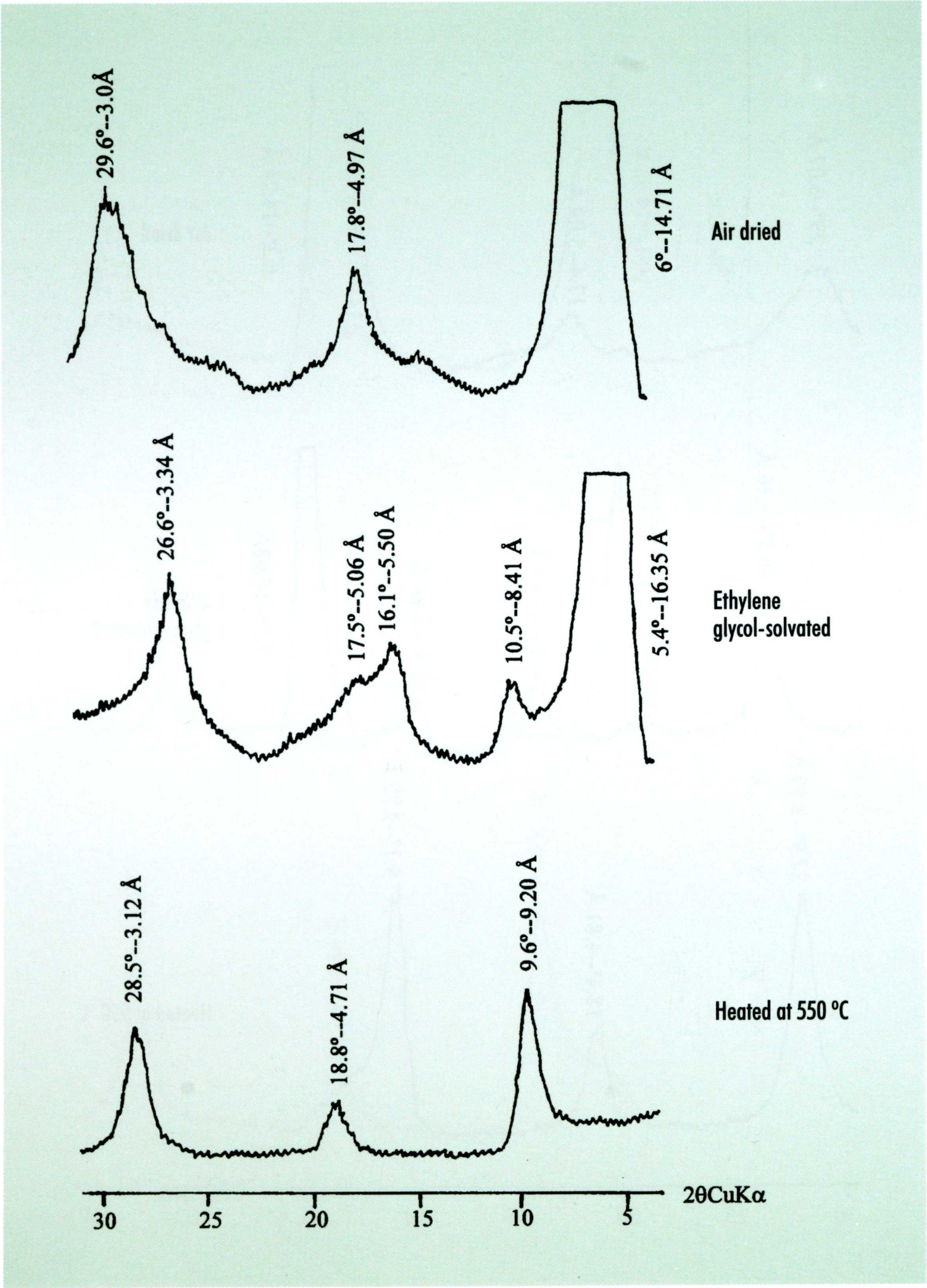


Figure 4.13: X-ray diffraction pattern (XRD) of the sample F-65 (oriented aggregate of the fraction less than 2 μm).

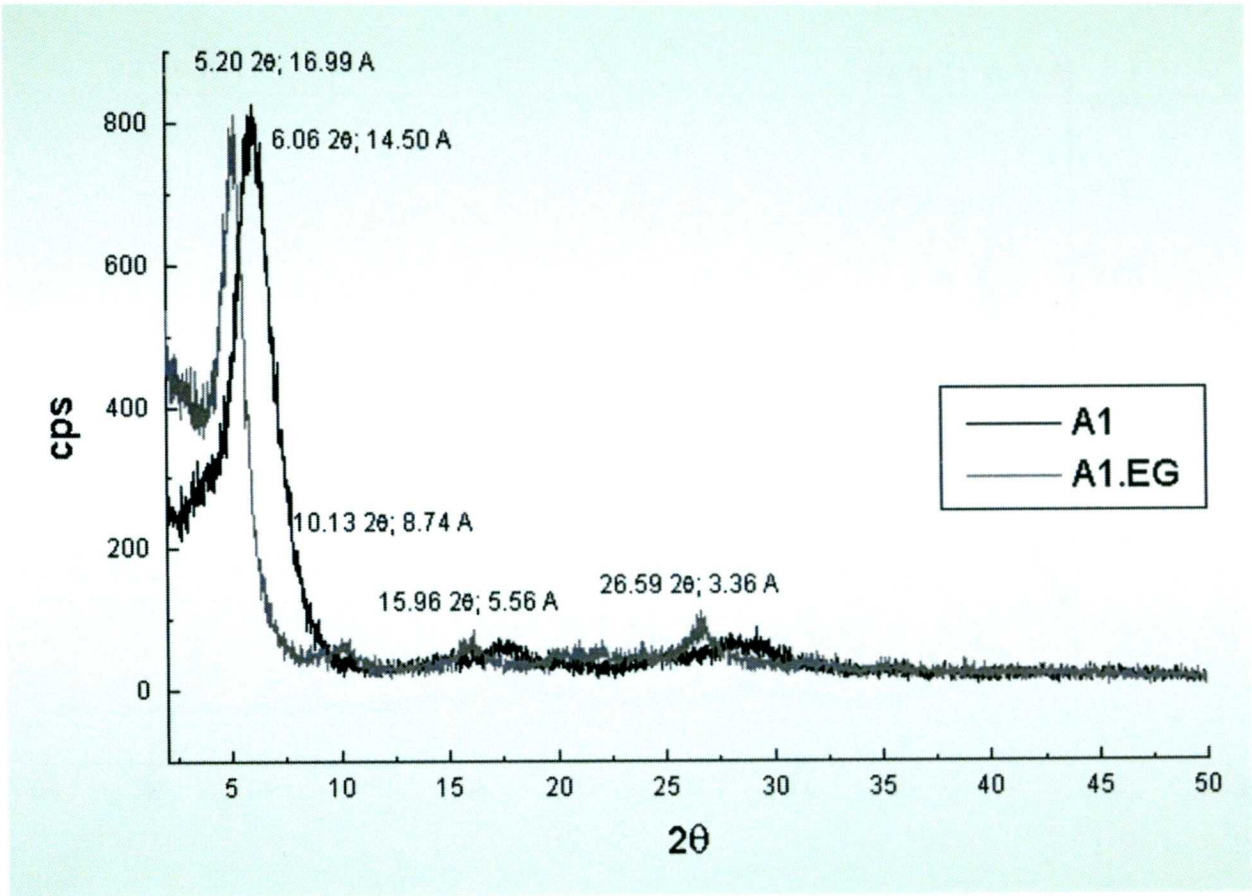


Figure 4.14: X-ray diffraction pattern (XRD) of the sample A-1 performed at UAM (normal and glycolated oriented aggregate of the fraction less than $2\ \mu\text{m}$).

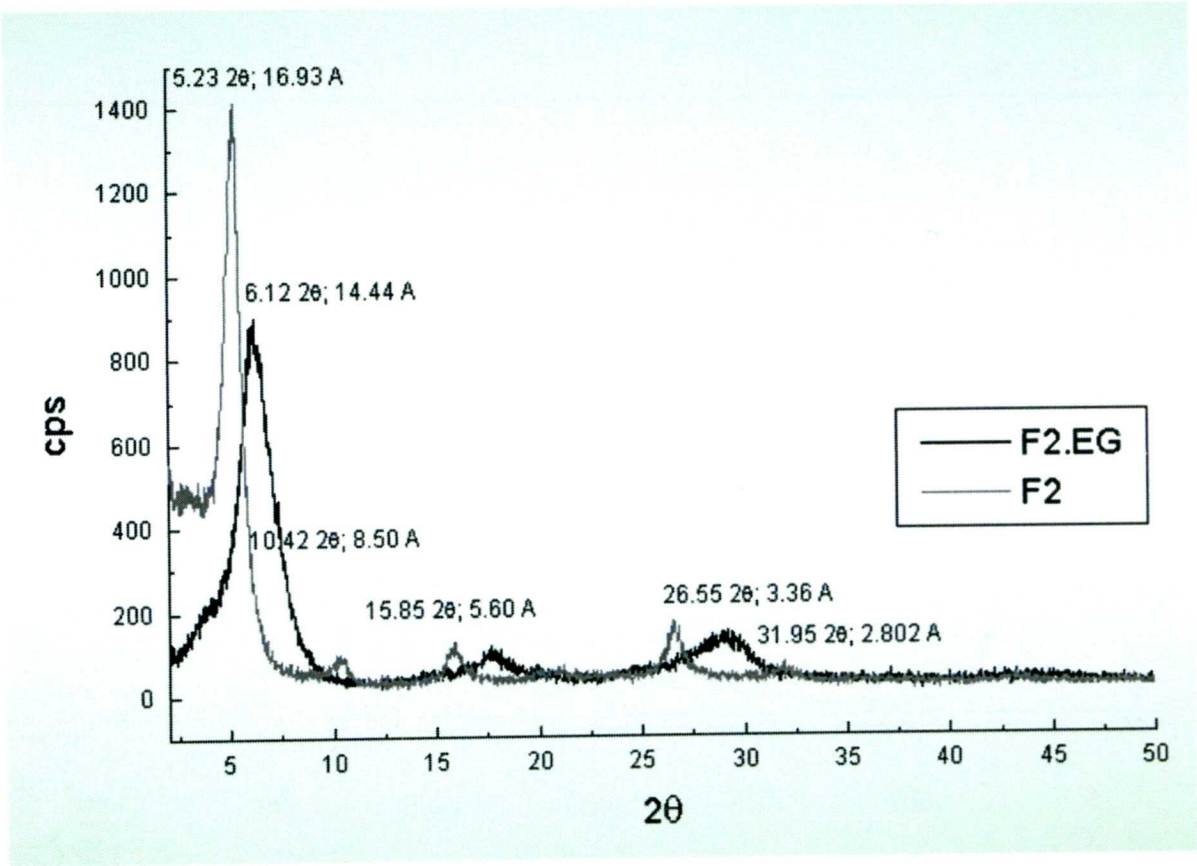


Figure 4.15: X-ray diffraction pattern (XRD) of the sample F-2 performed at UAM (normal and glycolated oriented aggregate of the fraction less than $2\ \mu\text{m}$).

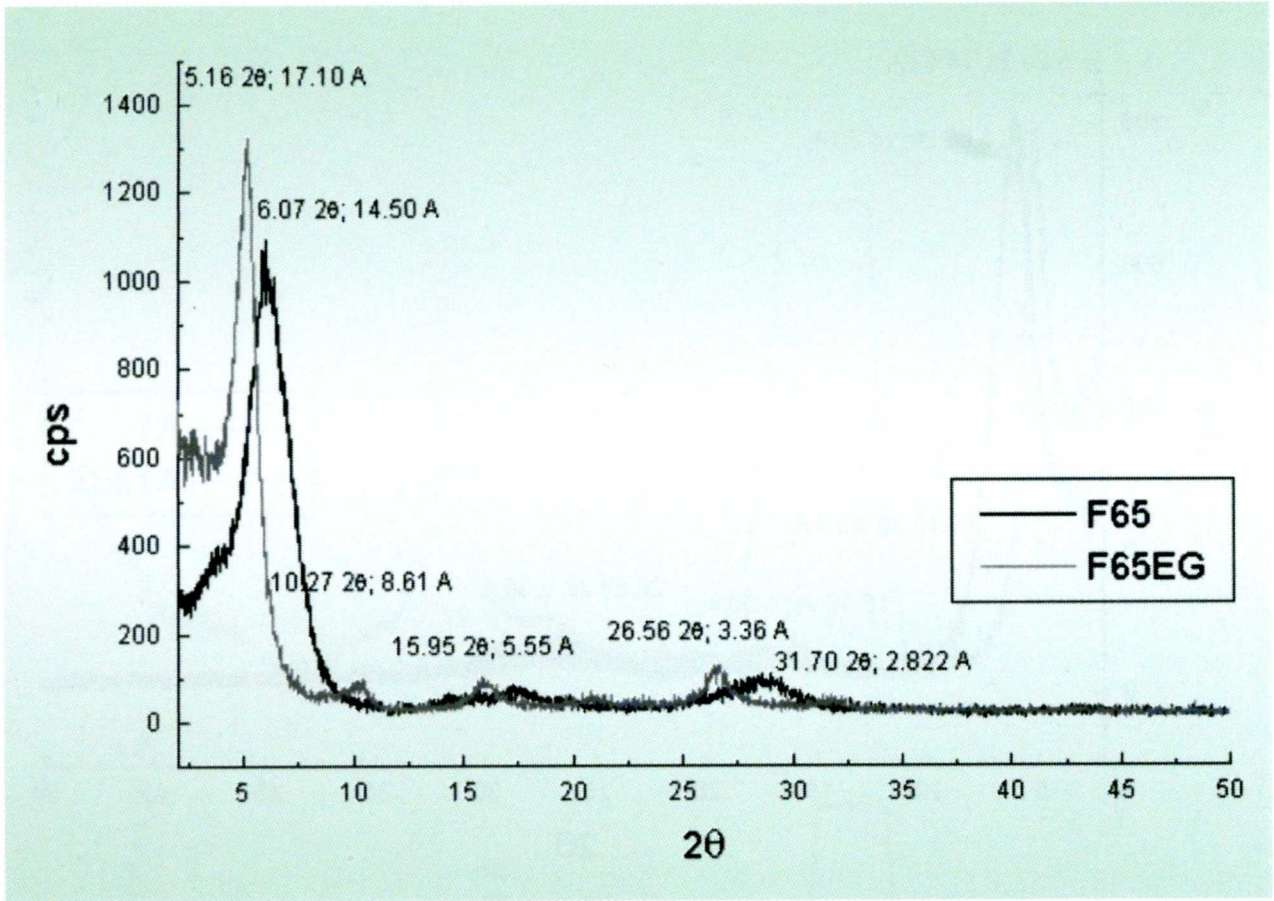


Figure 4.16: X-ray diffraction pattern (XRD) of the sample F-65 performed at UAM (normal and glycolated oriented aggregate of the fraction less than 2 μm).

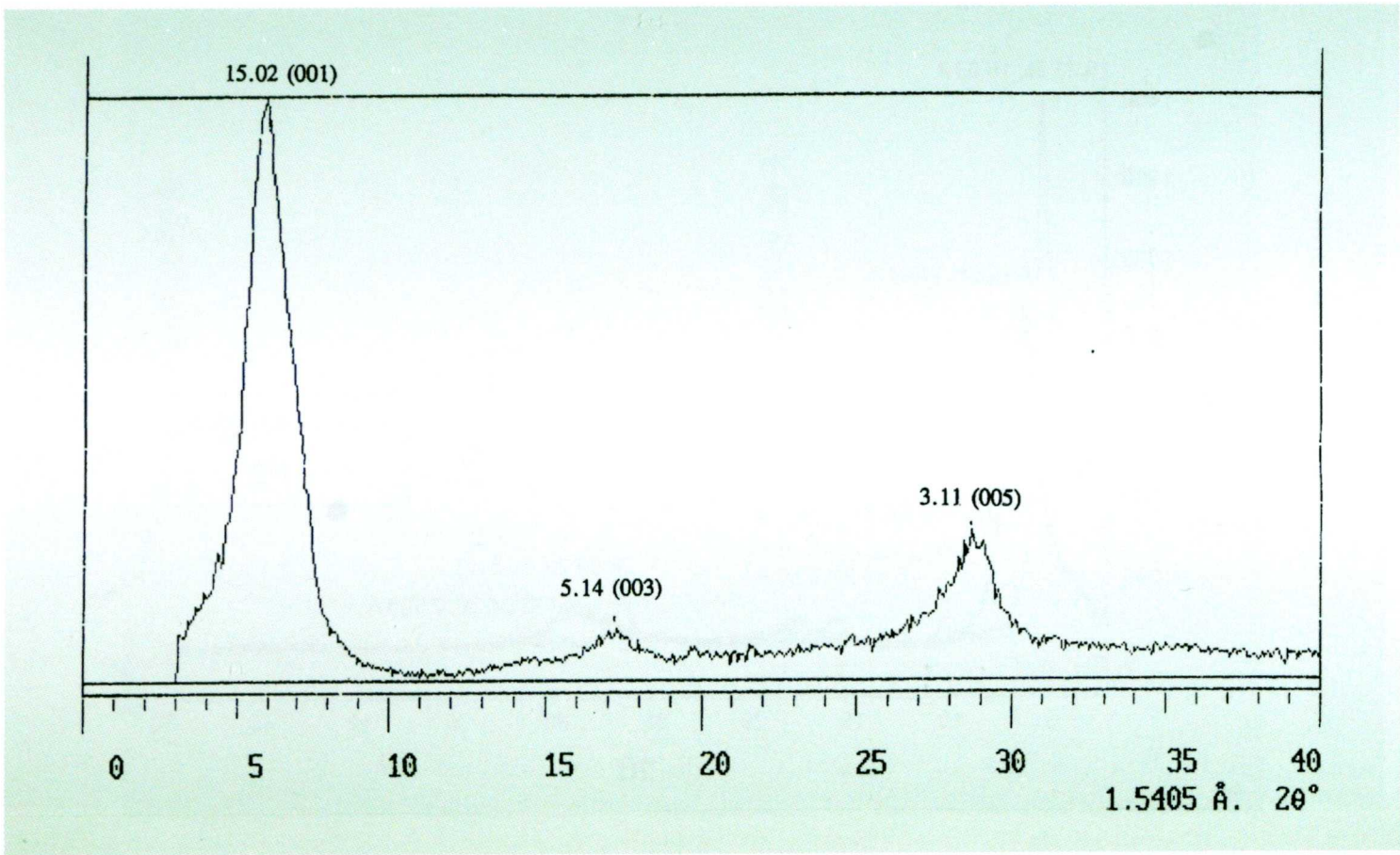


Figure 4.17: X-ray diffraction pattern (XRD) of the sample E-1 (oriented aggregate of the fraction less than 2 μm).

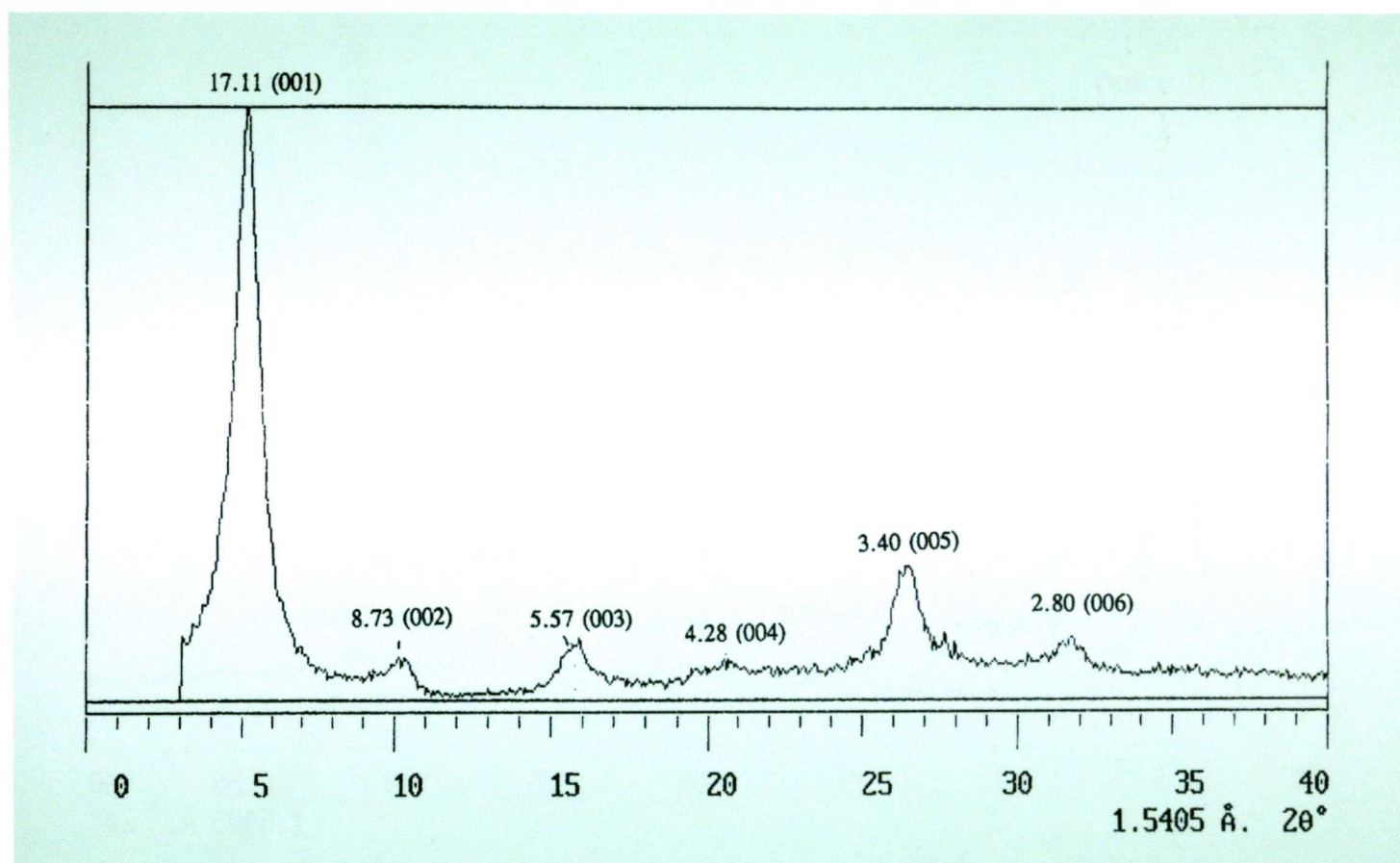


Figure 4.18: X-ray diffraction pattern (XRD) of the sample E-1 (oriented aggregate and ethylene glycol of the fraction less than $2 \mu\text{m}$).

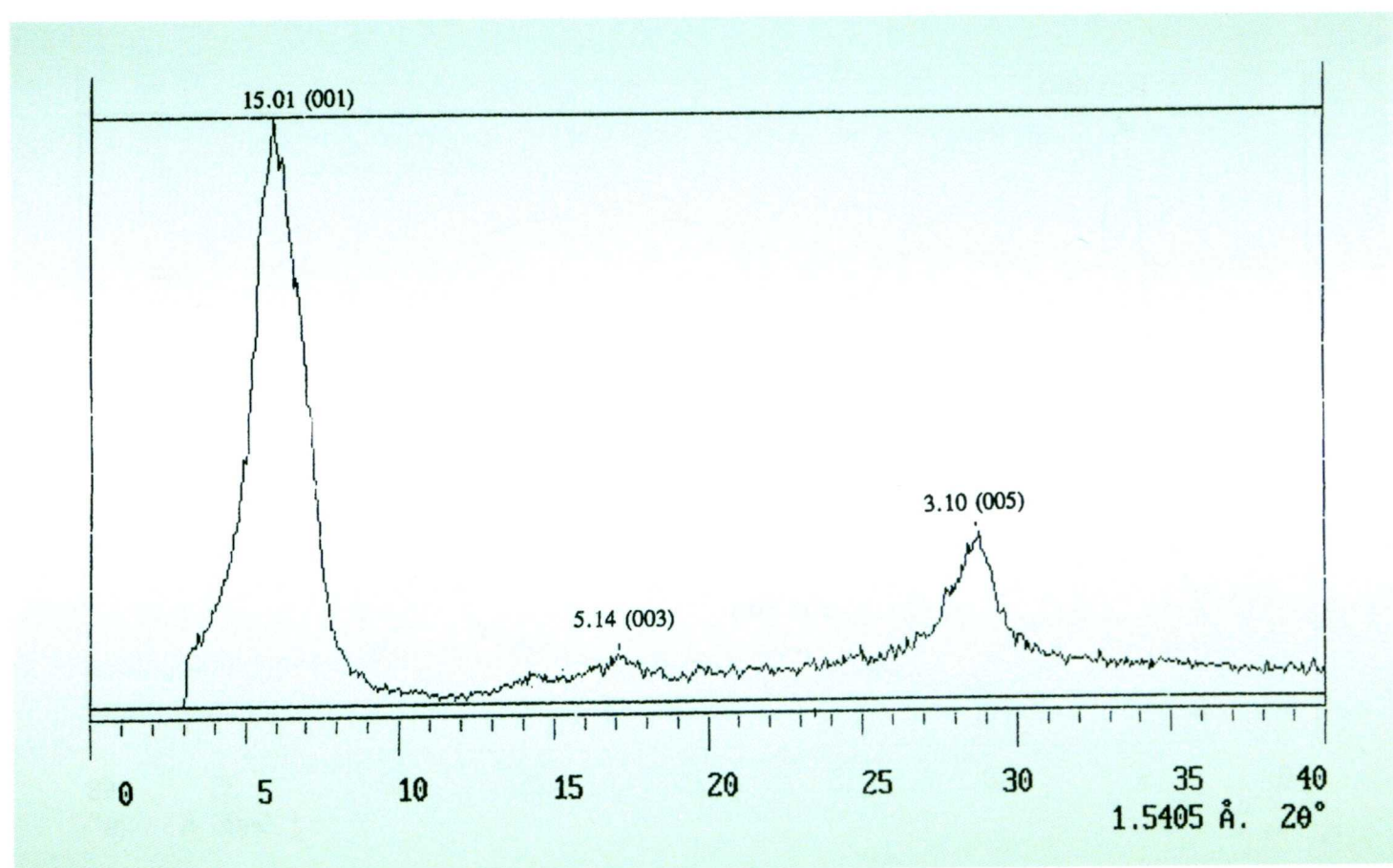


Figure 4.19: X-ray diffraction pattern (XRD) of the sample E-2 (oriented aggregate of the fraction less than $2 \mu\text{m}$).

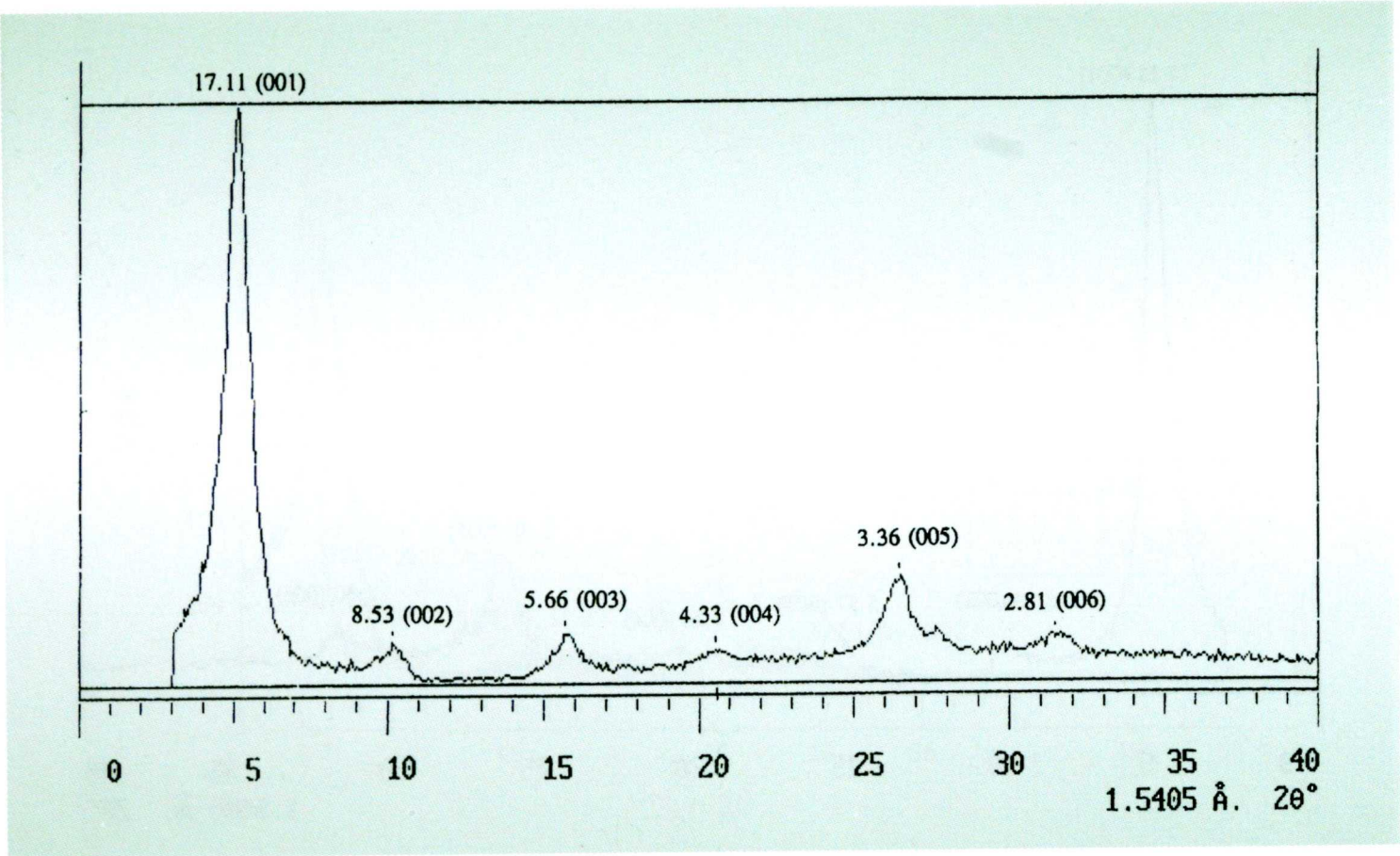


Figure 4.20: X-ray diffraction pattern (XRD) of the sample E-2 (oriented aggregate and ethylene glycol of the fraction less than $2 \mu\text{m}$).

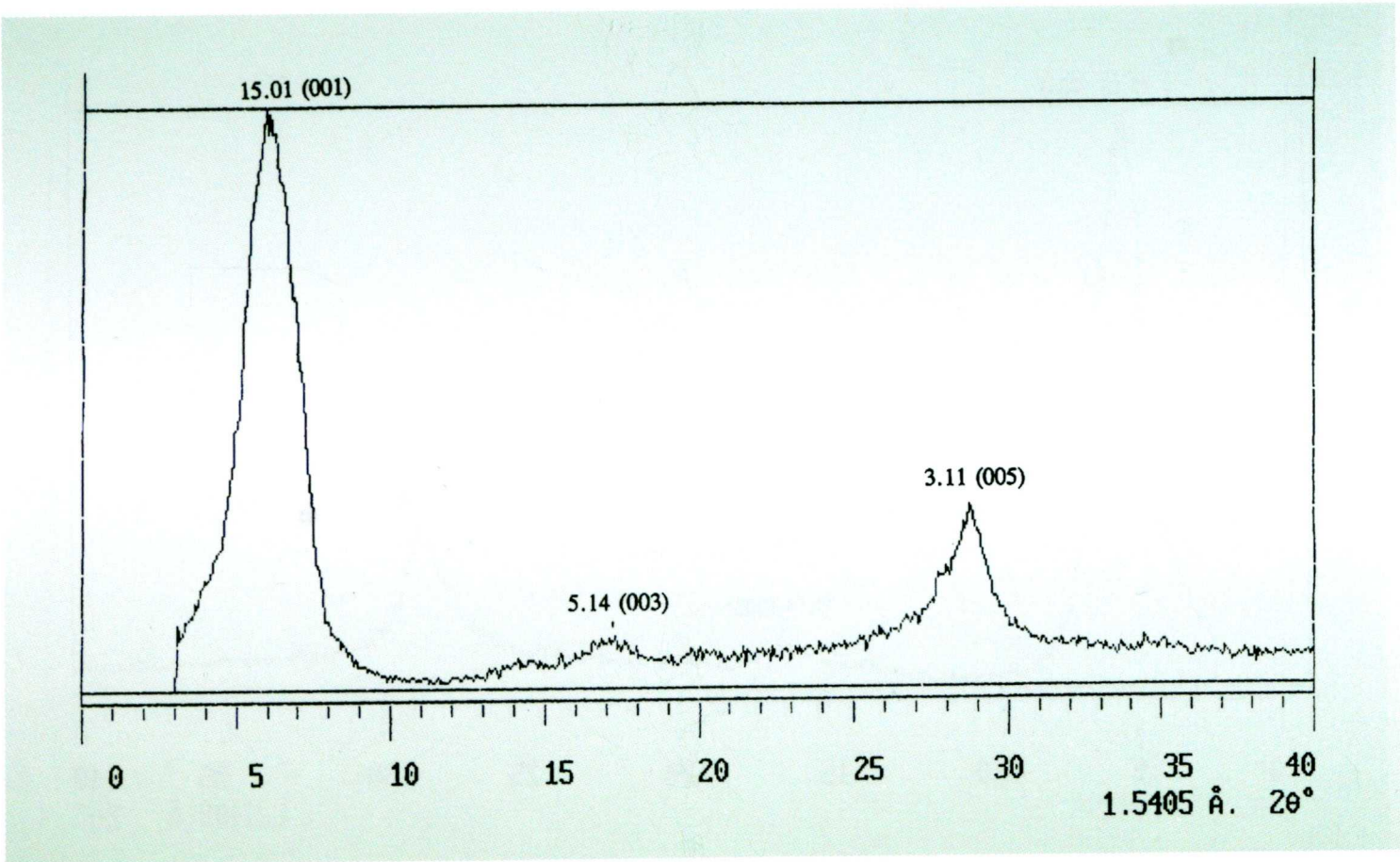


Figure 4.21: X-ray diffraction pattern (XRD) of the sample E-3 (oriented aggregate of the fraction less than $2 \mu\text{m}$).

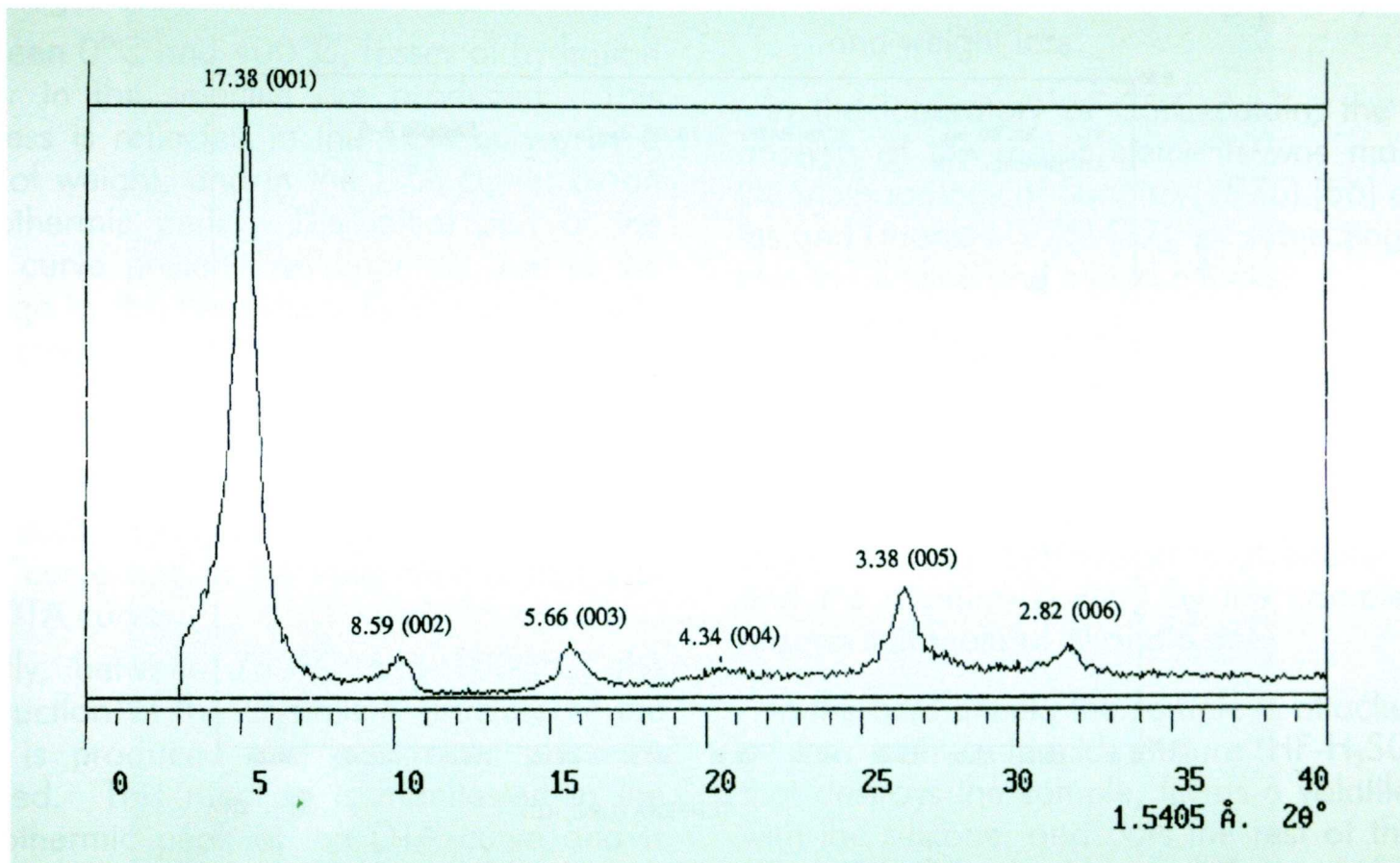


Figure 4.22: X-ray diffraction pattern (XRD) of the sample E-3 (oriented aggregate and ethylene glycol of the fraction less than $2 \mu\text{m}$).

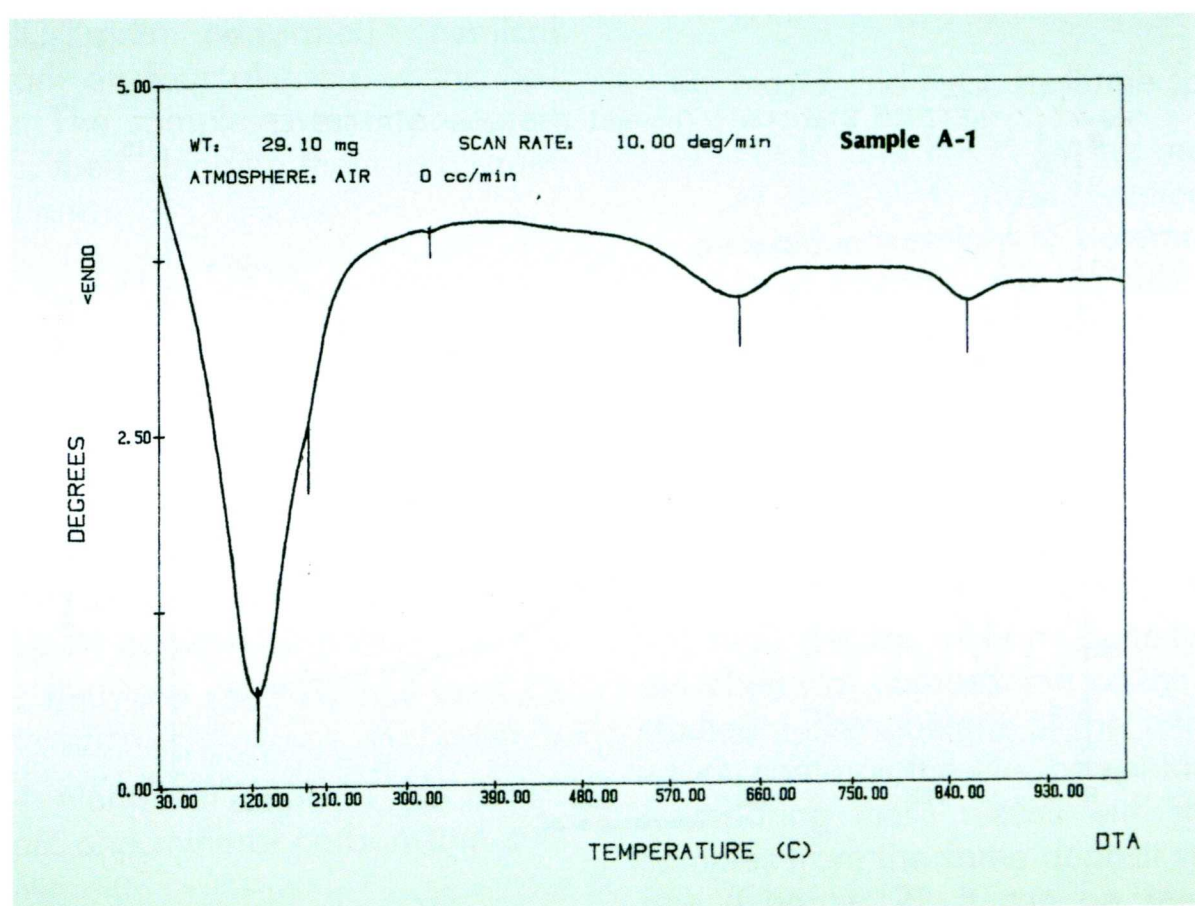


Figure 4.23: DTA curve of sample A-1.

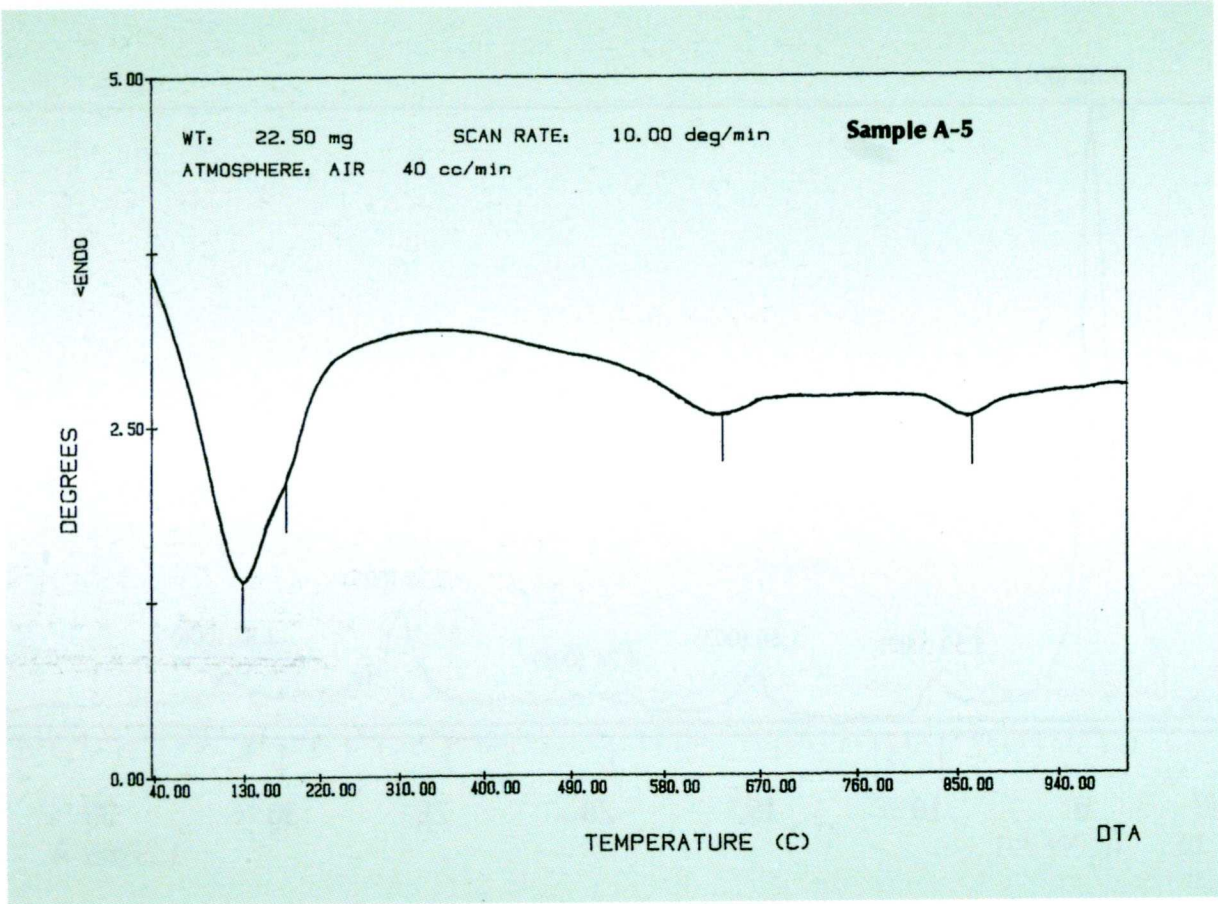


Figure 4.24: DTA curve of sample A-5.

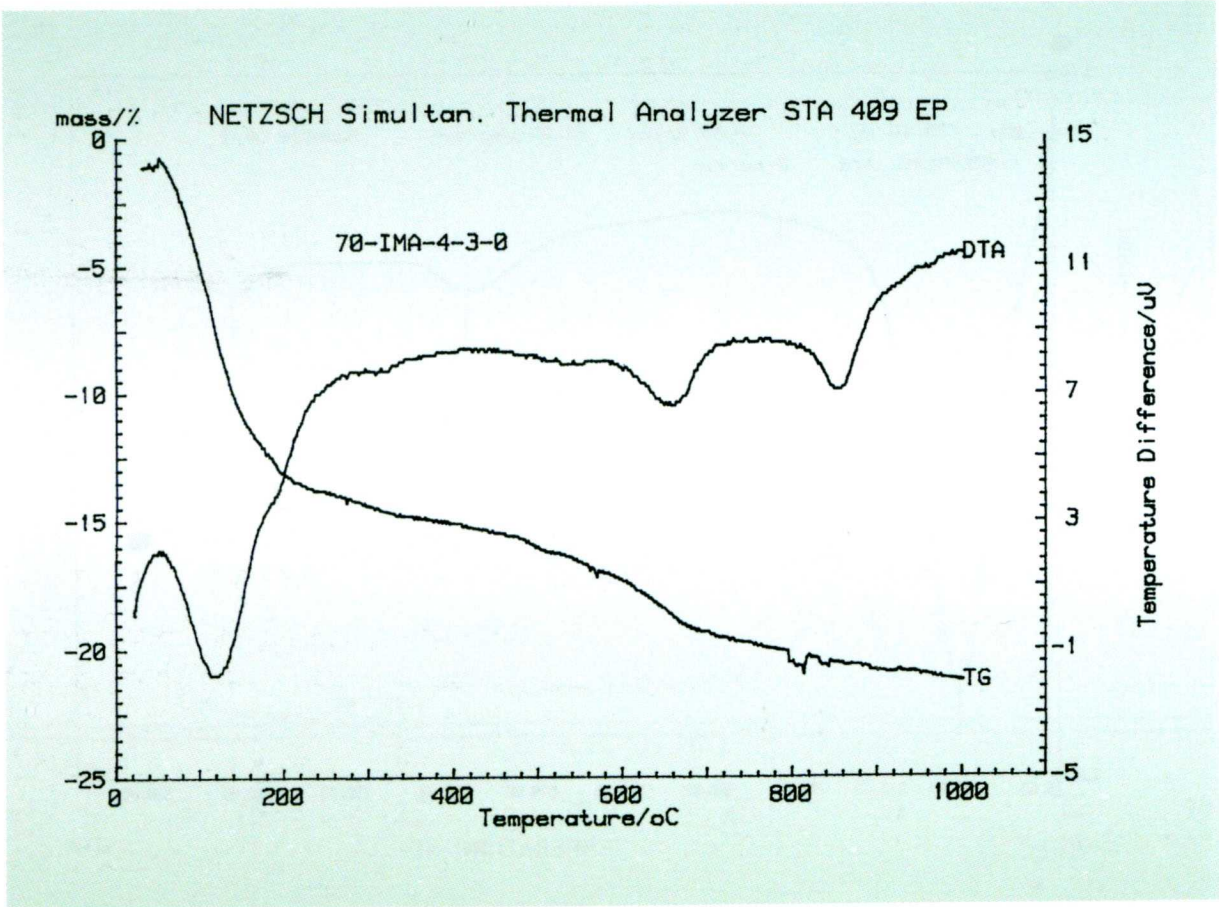


Figure 4.25: DTA and TGA curves of sample 70-IMA-3-4-0.

The patterns obtained are typical of a smectite, and three stages are differentiated therein:

- Between 0°C and 400°C, losses of hydration water in the smectite are produced. This process is reflected in the TGA curve, by a loss of weight, and in the DTA curve, by an endothermic peak. The initial part of the DTA curve presents an anomaly due to the change in the heat capacity of the clay with an increasing temperature.
- Between 400°C and 750°C, the loss of structural water in the clay (hydroxyls in the octahedral layer) is produced and that manifests itself equally in the loss of weight in the TGA curve and in the endothermic peaks of the DTA curve.
- Finally, between 750°C and 1000°C, the destruction of the crystalline structure of the clay is produced and a vitreous phase is formed. This reaction is manifested in the endothermic peak of the DTA curve and is not manifested in that of the TGA, as a change of mass has not occurred.

4.3.2. Chemical characterization

The chemical characterization was performed by the CIEMAT laboratory on four unmixed samples (F samples) and on two of the homogenized sample (A samples); CSIC-Zaidín performed chemical characterization work on four aliquots of the homogenized sample. The samples were prepared by drying at 60°C, then grinding them to a grain size of less than 60 µm.

Methods

- Inductively coupled plasma-atomic emission spectrometry for Al_2O_3 , Fe_2O_3 total, Na_2O , MgO , MnO , TiO_2 , CaO , P_2O_5 , Ba, Ce, Co, Cr, Cu, La, Ni, Sr, V, Y, and Zn.
- X-ray fluorescence for SiO_2 , on beads, and Rb, Th, and U, on powdery samples.
- Flame atomic emission spectroscopy for K_2O and Li.
- Leco CS-244 elemental analyzer for determining organic and mineral carbon and sulfur.
- Sánchez-Ledesma et al (1990) [55] method for FeO.
- Selective ion electrode for Cl^- and F^- .

- Thermogravimetric analysis (TGA), using a Perkin-Elmer TGS-2 equipment, for H_2O^- and weight loss.

In the laboratory of CSIC-Zaidín, the chemical analysis of the major elements was made, using the methodology of Shapiro (1975) [56] and Huer-tas and Linares (1975) [57], by subjecting the samples to alkaline and triacid attacks.

In the basic attack, the sample was fused with NaOH. By this method, the bentonite was destroyed and the silica and alumina passed into solution, where they were determined by ultraviolet-visible spectrophotometry. The silica content was determined by the silicon molybdenum method and the alumina content by the complex sodium alizarin sulfonate of Al and Ca.

In the acid attack, the sample is attacked in a digestion with a triacid mixture $\text{HF-H}_2\text{SO}_4\text{-HNO}_3$, that destroys the sample, forms a volatile complex with the silicone, and puts the rest of the components (Fe, Ca, Mg, Na, and K) into solution. The determinations were made in the following form:

- Fe_2O_3 , in ultraviolet-visible spectrophotometry, by the method of formation of the complex with 1,10-phenanthroline after its reduction with hydroxylamine chlorhydrate.
- CaO and MgO, volumetrically by EDTA titration.
- Na_2O and K_2O , by flame spectrophotometry.
- H_2O^+ and H_2O^- , by the method of Cuadros et al (1994) [58]: heating the sample to 300°C overnight to determine the H_2O^- and later heating the sample to 1000°C for about two hours to determine the H_2O^+ .

Major elements — Results

The results of the chemical analyses of the major elements are presented in Table 4.5.

A high degree of homogeneity was observed in the chemical composition of the different samples studied. The content of the mineral CO_2 is low, which confirms the low percentage of carbonates. Comparing these results with those obtained on samples from the same deposit studied prior to FEBEX (Chapter 2), it can be seen that the FEBEX bentonite has lower contents of Fe_2O_3 , MgO , CaO , and organic CO_2 . The lower content of Fe_2O_3 could explain the lower specific weight (Sec-

Table 4.5
Chemical composition of bulk sample (CIEMAT)

Oxides %	F-2	F-37	F-75	F-119	AVERAGE %	STD. DEV.	A-1	A-5
SiO ₂	58.6	59.4	59.2	60.5	59.4	0.8	59.9	58.7
Al ₂ O ₃	18.2	18.4	17.8	18.1	18.1	0.3	16.6	17.9
Fe ₂ O ₃	2.93	2.96	2.96	2.87	2.93	0.04	2.66	2.90
FeO	0.24	0.22	0.22	0.21	0.21	0.02	0.22	0.18
MgO	4.2	4.3	4.1	4.2	4.2	0.1	3.9	4.2
MnO	0.04	0.04	0.04	0.04	0.04	0.00	0.04	0.04
CaO	1.8	1.9	2.0	1.8	1.9	0.1	1.7	1.8
Na ₂ O	1.3	1.4	1.4	1.4	1.4	0.1	1.2	1.3
K ₂ O	1.1	1.1	1.1	1.0	1.1	0.1	1.0	1.0
TiO ₂	0.23	0.24	0.25	0.23	0.24	0.01	0.22	0.23
P ₂ O ₅	0.02	0.03	0.02	0.03	0.02	0.00	0.02	0.02
H ₂ O ⁻	7.5	6.0	7.6	6.0	6.8	0.9	12.0	7.0
H ₂ O ⁺	4.4	4.6	4.8	4.9	4.7	0.2	4.0	4.8
CO ₂ org	0.40	0.37	0.40	0.33	0.37	0.03	0.29	0.33
CO ₂ min	0.26	0.22	0.26	0.22	0.24	0.02	0.37	0.26
SO ₂ total	0.22	0.10	0.16	0.16	0.16	0.05	0.36	0.12
F ⁻	0.18	0.18	0.18	0.19	0.18	0.01	0.18	0.18
F ≡ O	0.08	0.08	0.08	0.08	0.08	0.00	0.08	0.08

tion 4.2.5) of the FEBEX bentonite than that found in the previous studies.

The average value for the results of the chemical analyses performed on four samples in the laboratory of CSIC-Zaidín are shown in Table 4.6 and those values are typical of a smectite with a silica content close to 60 %.

The small differences between the results from the two laboratories can be expected considering

the variations in the material and in the methods used for preparation and testing.

Trace elements — Results

The results of the chemical analyses of the trace elements in the bulk sample are presented in Table 4.7.

The content of the trace elements is similar to that determined in the studies prior to FEBEX on the

Table 4.6
Chemical analysis (CSIC-Zaidín)

Reference	SiO ₂ %	Al ₂ O ₃ %	Fe ₂ O ₃ %	CaO %	MgO %	Na ₂ O %	K ₂ O %	H ₂ O %	Total %	H ₂ O ⁻ %
AVERAGE	58.92	19.48	3.48	2.51	4.83	2.28	1.21	7.09	99.82	5.07
STD. DEV	0.03	0.31	0.08	0.29	0.15	0.09	0.10	0.18	0.08	0.12

bentonite from the same deposit (Chapter 2), except for Cu, which was not detected in the previous studies, and for Cr, which is higher in the FEBEX bentonite.

4.3.3. Structural formula

The chemical analyses of the fraction less than 2 μm were made on samples A-1 and A-5 (homogenized sample), after their saturation with calcium to homoionize the cation exchange complex. In this way, all the cations are displaced from the interlayer, with this the Mg detected in the chemical analysis can be attributed to the octahedral layer (Wilson, 1987) [59].

The results are presented in Table 4.8.

The structural formula has been calculated considering that the anionic charge is -44, with an ideal composition of the anion O₂₀(OH,F,Cl)₄. The structural formulas of the smectites corresponding to samples A-1 and A-5 are presented in Table 4.9.

The smectite of sample A-1 has an octahedral charge of -1.02, a tetrahedral charge of -0.15 (15% of the octahedral), and the interlayer charge is 1.15. For the smectite of sample A-5 these values are, respectively, -0.96, -0.20 (17% of the octahedral charge) and 1.18.

The smectite of both samples has an aluminic dioctahedral layer structure, with Mg in octahedral sites, and with 15% of the charge in sample A-1 and 17% of the charge in sample A-5, both of which were caused by tetrahedral substitution. Consequently, these smectites are Wyoming-type montmorillonites (according to L.G. Schultz, in A. C. D. Newman, 1987) [60].

The cation exchange capacity (CEC) of the fraction less than 2 μm has been calculated, for the samples A-1 and A-5, according to the following method:

$$CEC \text{ (meq/100 g)} = (1000 \cdot \%CaO \cdot \text{valence}) / P_m \text{ of the oxide}$$

The results are 127 meq/100 g for sample A-1 and 125 meq/100 g for A-5, which are very high.

In the tests made in the laboratory of CSIC-Zaidín, the exchange capacity was determined by means of saturation with ammonium acetate, at pH 7, and by later displacement of the NH₄⁺ using the Kjeldhal method.

Four samples were tested with three measurements made on each. The values are high, with an average of 106 meq/100 g, and correspond to an exchange capacity of a bentonite which is very rich in smectite.

The structural formula of smectite was calculated with the values obtained in the chemical analysis and the exchange capacities made on the fraction less than 20 μm, which contains more than 99 % smectite. The structural formula obtained is:



4.3.4. Soluble salts, conductivity, pH, and amorphous phases

The content of soluble salts has been determined by the CIEMAT laboratory, following Procedure 70-IMA-G-0-29 [61], on 7 unmixed samples (F samples) and on 2 of the homogenized sample (A samples).

A dry sample of 2.5 g was dispersed in 10 cm³ of distilled water (relation solid to liquid 1:4) and stirred for 48 hours. Later the suspension was centrifuged and, in the supernatant, the chlorides and sulphates were determined by ion chromatography and the electric conductivity by an electrometric cell. The salt content does not appear in the X-ray determination and, therefore, is always under 1%.

Table 4.7
Trace elements in bulk sample (CIEMAT)

Element ppm	F-2	F-37	F-75	F-119	Average ± Std. Deviation	A-1	A-5
B _{tot}							
¹⁰ B/ ¹¹ B	0.2500	0.2506	0.2466		0.249±0.002	0.2453	0.2453
Ba	135	180	190	160	166±24	150	195
Be	<5	<5	<5	<5	<5	<5	<5
Br	2.2	2.3	2.2	2.5	2.3±0.1	2.5	2.1
Ce	64	68	65	71	67±3	64	62
Cl ⁻	590	605	600	592	597±7	580	575
Co	15.0	8.2	8.3	8	9.9±3.4	8.3	8.7
Cr	7.2	7.9	5.9	6.4	6.8±0.9	11.0	6.4
Cs	10.0	11.0	9.8	9.6	10.1±0.6	10.0	10.0
Cu	20	28	14	39	25±11	30	31
Eu	0.76	0.79	0.79	0.83	0.79±0.03	0.75	0.75
I	<2	<2	<2	<2	<2	<2	<2
La	42	43	40	43	42±2	38	41
Li	55	58	55	55	56±2	54	54
Mo	<5	<5	<5	<5	<5	<5	<5
Nd	25	28	27	29	27±2	26	27
Ni	19	21	16	22	20±3	24	21
Rb	40	41	44	39	41±2	44	41
Re	<0.1	<0.1	<0.1	<0.1	<0.1	<0.1	<0.1
Sn	<10	<10	<10	<10	<10	<10	<10
Se	<1	<1	<1	<1	<1	<1	<1
Sr	215	225	245	250	224±17	190	220
Th	20.1	20.1	19.2	20.1	19.9±0.5	19.0	19.7
U	1.5	2.6	1.3	2.2	1.9±0.6	1.9	1.9
V	16	16	19	16	17±2	12	14
W	<25	<25	<25	<25	<25	<25	<25
Y	20	24	23	25	23±2	23	22
Zn	62	71	60	69	66±5	64	70

Table 4.8
Chemical composition of fraction less than 2 μm homoionized in Ca

Oxide %	A-1	A-5	Average %
SiO ₂	60.51	60.17	60.34
Al ₂ O ₃	19.14	19.41	19.27
Fe ₂ O ₃	3.26	3.46	3.36
FeO	0.17	0.14	0.155
MgO	4.04	4.22	4.13
CaO	3.57	3.50	3.53
Na ₂ O	0.28	0.35	0.31
K ₂ O	0.61	0.67	0.64
Ti ₂ O	0.18	0.17	0.17
H ₂ O ⁺	8.25	7.90	8.07

The free silica was extracted in a 0.1 M solution of Na₂CO₃ after 48 hours of stirring at ambient temperature (Procedure 70-IMA-G-0-33 [62]). Later the suspension was centrifuged and the free silica measured in the supernatant by the silico-molybdc colorimetric method.

The values obtained are shown in Tables 4.10 and 4.11.

In comparing the results of the present study with the values obtained during the prior studies (Chapter 2), it can be observed that the average value of the chlorides is less and the average content of free silica is greater for the FEBEX bentonite than

for the previously-studied bentonite from the same deposit.

In the laboratory of CSIC-Zaidín, the soluble cations, the pH, and the electric conductivity were determined in an aqueous extract made with distilled water in a relation of solid to liquid 1:10.

The ions Na⁺, K⁺, Ca²⁺, and Mg²⁺ were determined. The average values obtained were: 133.66 mg/100 g of Na⁺; 6.01 mg/100 g of K⁺, 6.45 mg/100 g of Ca²⁺, and 1.12 mg/100 g of Mg²⁺. The electric conductivity of the aqueous extract was measured by comparing it with that of a standard solution: the average value obtained was 0.59 mS/cm. The pH of the aqueous extract was meas-

Table 4.9
Structural formula of the smectite

Sample	Tetrahedral		Octahedral					Interlayer		
	Si ⁴⁺	Al ³⁺	Al ³⁺	Fe ³⁺	Fe ²⁺	Ti ⁺	Mg ²⁺	Ca ²⁺	Na ⁺	K ⁺
A-1	7.85	0.15	2.78	0.32	0.02	0.02	0.78	0.49	0.07	0.10
A-5	7.80	0.20	2.76	0.34	0.01	0.02	0.82	0.49	0.09	0.11

Table 4.10
Analysis of aqueous extract 1:4

Sample reference	E.C. ^{25°C} μS/cm	Cl ⁻ mg/l	SO ₄ ²⁻ mg/l
F-2	1 512	200	294
F-17	1 635	192	336
F-18	1 557	193	303
F-37	1 423	182	258
F-75	1 579	203	309
F-102	1 468	184	299
F-119	1 412	200	200
AVERAGE	1 512	193	286
STD. DEV.	83	8	44
A-1	1 601	197	319
A-5	1 557	193	303

ured by the usual method, adjusting the pH-meter with two standards of 4 and 7 units of pH; the average pH was alkaline and varied between 8.98 and 9.00.

CSIC-Zaidín also obtained the amorphous phases of silica, iron, and alumina in their laboratory.

To extract the amorphous phases of silica and alumina, a 5 % sodic carbonate solution was used as an extractor. The extracted solution was made acidic, to a pH of 2.5, with HCl, and the determination of the free silica and free alumina were made by spectrophotometry with the previously-described methods.

The extraction of the iron oxyhydroxides was made with sodic dithionite and sodic citrate following the method of Holgrem (1967) [63]. Once extracted, the iron was determined by spectrophotometry with the method previously described.

The average values obtained for these gels are indicated in Table 4.12.

4.3.5. Cation exchange complex

Once the soluble salts were leached, the exchangeable cations of clay were extracted by successive displacements with a buffered solution 1 M of AcONH₄ (ammonium acetate) of pH 7 (Procedure 70-IMA-G-0-34 [64]) and were determined by atomic emission spectroscopy, except that K⁺ was determined by flame emission. The results from each sample are shown in Table 4.13, together with the value of the CEC obtained by adding the value of each cation.

All the samples have small quantities of other exchangeable cations: 0.41 meq/100 g of Sr²⁺, 0.02 meq/100 g of Ba²⁺, and 0.06 meq/100 g of Mn²⁺; some samples also contain small quantities (less than 0.4 meq/100 g) of Al³⁺.

In the CSIC-Zaidín laboratory, the exchangeable cations were determined together with the cation exchange capacity, following the procedure indicated in Section 4.3.3.

The exchangeable cations were determined in the solutions after the initial saturation with NH₄⁺:

Table 4.11
Free silica in solid phase

Sample reference	Free SiO ₂ ppm
F-2	379
F-17	338
F-18	320
F-37	398
F-75	316
F-102	433
F-119	486
AVERAGE	381
STD. DEV.	63
A-1	419
A-5	408

Table 4.12
Amorphous phases

	Amorphous phases		
	% SiO ₂	% Al ₂ O ₃	% Fe ₂ O ₃
AVERAGE	1.48	0.035	0.105
STD. DEV.	0.11	5 · 10 ⁻³	8.6 · 10 ⁻³

Na and K by flame photometry and Ca and Mg by EDTA titration. The average values obtained are shown in Table 4.14. The exchange complex is dominated by Mg²⁺, Ca²⁺, and Na⁺, but always having a minor content of K⁺.

The values obtained by the two laboratories, for the cation exchange complex, are very similar.

4.3.6. Total specific surface

The total specific surface was determined in the CSIC-Zaidín laboratory by the hygroscopicity

method (Keeling, 1961) [65]. Four aliquots of the sample were heated to 300°C for two hours and later cooled in a desiccator with CaCl₂.

The dried samples remained, until a constant weight was reached, in a desiccator that contained a saturated solution of NaCl, which produced a water vapor atmosphere of 80 %. Comparing the adsorbed water content with that of a standard Wyoming montmorillonite, the surficial area was calculated. The results varied between a maximum of 659 m²/g and a minimum of 640 m²/g.

4.4. Mechanical, hydraulic, and thermal tests

4.4.1. Mechanical tests

Swelling pressure

The swelling pressure has been determined in oedometers following Procedure 70-IMA-G-0-21 [37]. The tests were made on 11 unmixed samples (F samples) and on 5 of the homogenized sample (A samples), at two nominal dry densities: 1.60 g/cm³ and 1.70 g/cm³. In total, there were 22

specimens tested at a nominal dry density of 1.60 g/cm³ and 10 at a nominal dry density of 1.70 g/cm³.

The specimens were uniaxially compacted in 1.20-cm high oedometer rings with a diameter of 4.93 cm (for the specimens with a dry density of 1.60 g/cm³) and a diameter of 3.57 cm (for the specimens with a dry density of 1.70 g/cm³). The rings are placed in the oedometer cell between two porous stones and in contact with water at the bottom. The specimen is kept at a constant volume during the test, by increasing the loads on the specimen and thereby impeding the swelling produced by the absorption of water. The swelling

Table 4.13
Average values of the cation exchange complex (CIEMAT)

Sample reference	Ca ²⁺ meq/100g	Mg ²⁺ meq/100g	Na ⁺ meq/100g	K ⁺ meq/100g	CEC meq/100g
F-2	47	39	26	2.3	114
F-17	48	37	25	2.3	113
F-18	44	37	25	2.2	108
F-37	55	41	26	2.2	124
F-75	47	35	23	2.2	108
F-102	52	36	25	2.3	116
F-119	48	35	25	2.3	110
AVERAGE	48	38	25	2.2	113
STD. DEV.	4	2	1	0.0	6
A-1	54	39	25	2.2	121
A-5	49	39	26	2.2	117

Table 4.14
Average values of the cation exchange complex (CSIC-Zaidín)

Reference	Ca ²⁺ meq/100g	Mg ²⁺ meq/100g	Na ⁺ meq/100g	K ⁺ meq/100g	Total meq/100g	CEC meq/100g
AVERAGE	47.03	35.5	25.8	2.29	110.7	106
STD. DEV.	2.07	2.37	0.51	0.08	1.43	3.4

pressure is defined as the load per unit of surface of the specimen after equilibrium has been reached, where equilibrium is defined as no deformation during a 24-hour period.

In addition to the swelling pressure, the initial and final water contents, as well as the dry density of each specimen were determined. The results are presented in Table 4.15. The average dry densities of all the tests in each set (all the F and A samples) were 1.60 g/cm³ and 1.71 g/cm³ with the corresponding swelling pressures of 49.7 kg/cm² and 98.5 kg/cm², respectively.

From the results of the tests made on the previously-studied bentonite from the same deposit (Chapter 2), it had been determined that the swelling pressure and the dry density are related by the equation

$$P_s = \exp (5,9 \rho_d - 7,9)$$

where P_s is the swelling pressure expressed in MPa and ρ_d is the dry density in g/cm³.

Using this equation, the swelling pressure for dry densities of 1.60 g/cm³ and 1.71 g/cm³ are 46.6 kg/cm² and 89.3 kg/cm², respectively. Thus, the

average values of the specimens obtained in the actual tests made on the FEBEX bentonite are slightly higher than those calculated: 6.7 % and 10.3 % for the dry densities of 1.60 g/cm³ and 1.71 g/cm³, respectively. These variations are very small, even for results of tests made on specimens from the same sample. Figure 4.26 shows the values obtained, together with the regression curve of the bentonite S-2.

Swelling under load

To determine the swelling produced under a vertical load, 7 tests have been made on specimens from the homogenized sample (A-1 to A-5). For all the testing, the specimens were prepared with a nominal dry density of 1.60 g/cm³. Tests were made on 4 specimens with a 0.5 MPa load and another 3 with a load of 0.9 MPa.

The specimens are prepared and placed in the oedometer, in the same way as for the swelling pressure. A predetermined vertical load is applied while at the same time the specimen is in contact with water at the bottom, permitting saturation. During the test, the swelling is noted periodically until stabilization is reached.

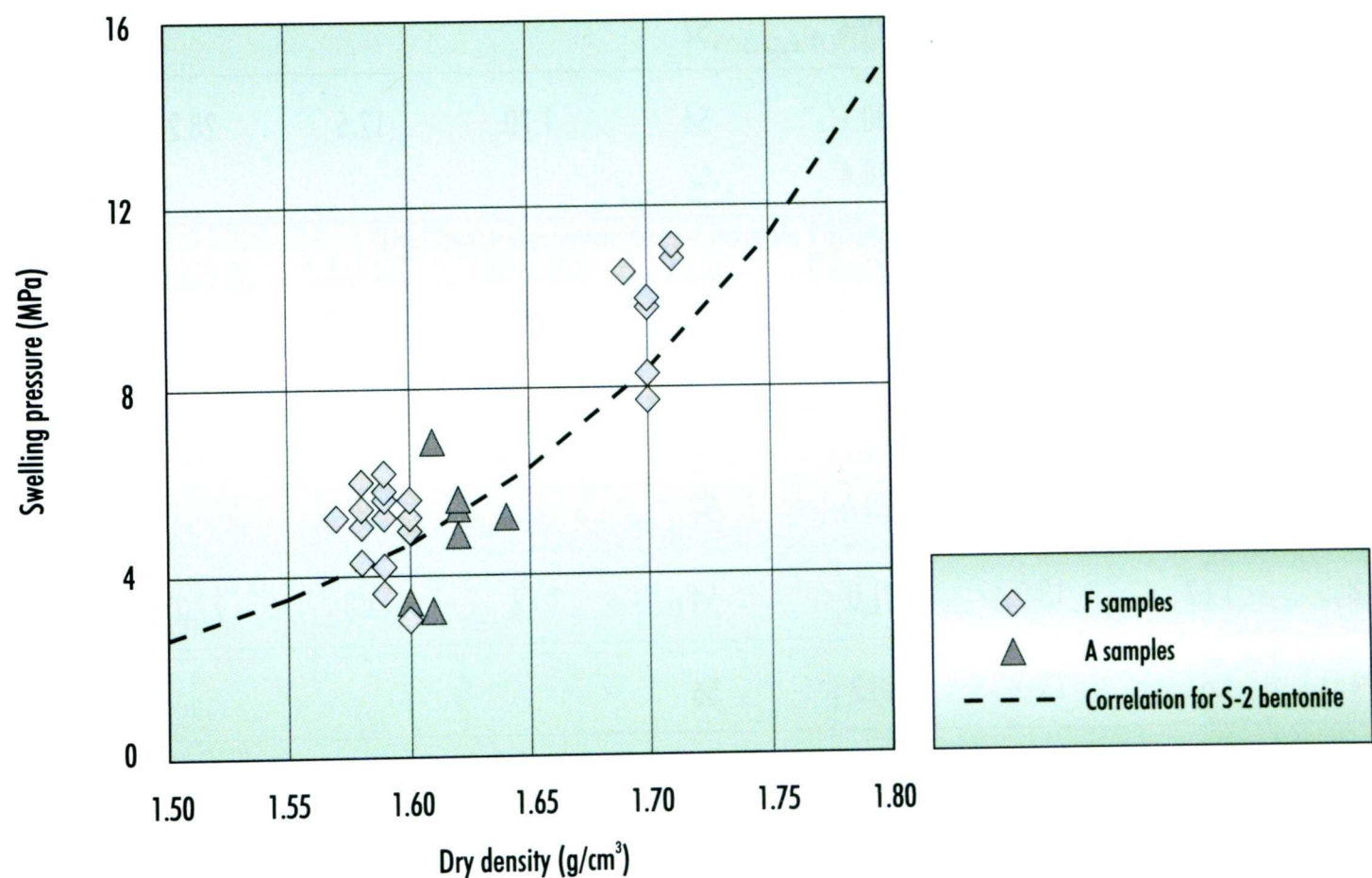


Figure 4.26: Swelling pressure for F and A samples and regression curve for bentonite S-2.

Table 4.15
Swelling pressure for nominal dry densities of 1.60 and 1.70 g/cm³

Sample reference	ρ_d g/cm ³	Initial w %	Final w %	P_s kg/cm ²	ρ_d g/cm ³	Initial w %	Final w %	P_s kg/cm ²
F-2	1.59	15.2	29.5	> 58	1.71	15.6	29.6	108
F-18	1.59	15.5	27.5	> 62	1.70	15.0	29.4	83
	1.58	16.1	29.4	> 50				
F-37	1.58	14.5	32.4	42	1.71	12.7	27.8	111
F-59	1.60	13.0	30.5	49				
F-65	1.58	15.0	30.1	> 60	1.70	12.7	28.4	77
	1.60	11.2	29.2	30				
F-75	1.59	12.2	29.2	52	1.70	12.3	27.5	97
	1.60	11.2	29.2	30				
F-79	1.59	13.1	32.5	41				
F-93	1.59	13.8	29.9	36	1.70	12.5	28.2	99
	1.59	14.0	29.7	> > 56				
F-102	1.60			> 56	1.69	14.8	28.4	105
F-109	1.57	14.3	29.6	52				
F-119	1.58	13.5	30.1	54	1.70	12.5	28.2	99
	1.60	12.5	28.4	52				
AVERAGE	1.59±0.01	13.9±1.4	29.9±1.3	50±9	1.70±0.01	13.4±1.4	28.5±0.8	97±13
A-1	1.61	13.0	29.9	32	1.72	13.4	29.0	85
	1.61	13.7	28.3	> 68				
A-2	1.64	12.0	26.7	52				
A-3	1.62	13.0	31.0	54	1.72	12.8	27.2	124
A-4	1.62	13.0	30.2	56				
A-5	1.60	14.5	31.9	34	1.72	13.4	28.2	96
	1.62	12.7	27.5	48				
AVERAGE	1.62±0.01	13.1±0.8	29.4±1.9	48±13	1.72±0.00	13.2±0.3	28.1±0.9	102±20

The swelling or strain percentage is the ratio between the increase in height and the initial height and is expressed as a percentage. The initial and final water contents as well as the initial dry density of each specimen are determined. The results are presented in Table 4.16.

Figure 4.27 shows the percentage of swelling as a function of time and it can be observed that stabilization is produced in 10 days.

Swelling tests under load were also made in the laboratory of UPC-DIT, following Procedure 70-UPC-G-0-02 [66]. Using material from the homogenized sample (70-IMA-3-4-0), which had been received from CIEMAT, 21 specimens were tested with various dry densities between 1.57 and 1.87 g/cm³ and with initial water contents of 11.7 ± 1.1% (water contents in equilibrium with the air in the laboratory). While being allowed to saturate, these specimens were tested under predetermined loads, between 0.01 MPa and 10.00 MPa, in conventional oedometers.

During saturation, vertical strains were measured with time. Figures 4.28 to 4.33 show, graphically, the evolution of the swelling strain as a function of time, for the different densities and vertical loads.

In Figures 4.34 and 4.35, the value, after saturation, of the vertical strain is represented as a function of the initial dry density and the applied vertical load.

The unit vertical strain (ϵ) induced by saturation can be expressed approximately as

$$\epsilon = - 0.4693 - 0.1935 \log \sigma + 0.3659 \rho_d$$

where σ is the vertical load in MPa and ρ_d is the initial dry density in g/cm³.

Figure 4.36 shows the volumetric change (expressed in void ratio) as a function of the load, after saturation. The resulting void ratio (e) can be expressed, approximately, by the equation:

$$e = 0.842 - 0.327 \log \sigma$$

where σ is the applied load in Mpa.

As can be seen, when compared, the results from both laboratories are similar.

State surface

These tests were made in the UPC-DIT laboratory on three specimens compacted to a dry density of 1.63 g/cm³ at a water content of 11.7 %. On each specimen, both a constant vertical load and a suction path were applied. The vertical loads, in MPa, were 0.03, 0.21, and 1.21. The suction path started with 130 MPa, which is the order of magnitude estimated for the initial water content,

Table 4.16
Swelling under vertical load (sample 70-IMA-3-4-0)

Aliquot	Vertical load 0.5 MPa				Vertical load 0.9 MPa			
	Initial ρ_d g/cm ³	Initial w %	Final w %	Swelling %	Initial ρ_d g/cm ³	Initial w %	Final w %	Swelling %
A-1	1.62	13.2	39.3	17.8				
A-2	1.60	14.7	40.1	17.2	1.59	14.2	36.7	15.9
A-3					1.61	13.8	36.6	14.7
A-4	1.62	13.7	38.3	17.8	1.59	15.0	37.2	13.4
A-5	1.62	14.0	39.1	17.2				
AVERAGE	1.61±0.01	13.9±0.6	39.2±0.7	17.5±0.3	1.60±0.01	14.3±0.6	36.8±0.3	14.7±1.3

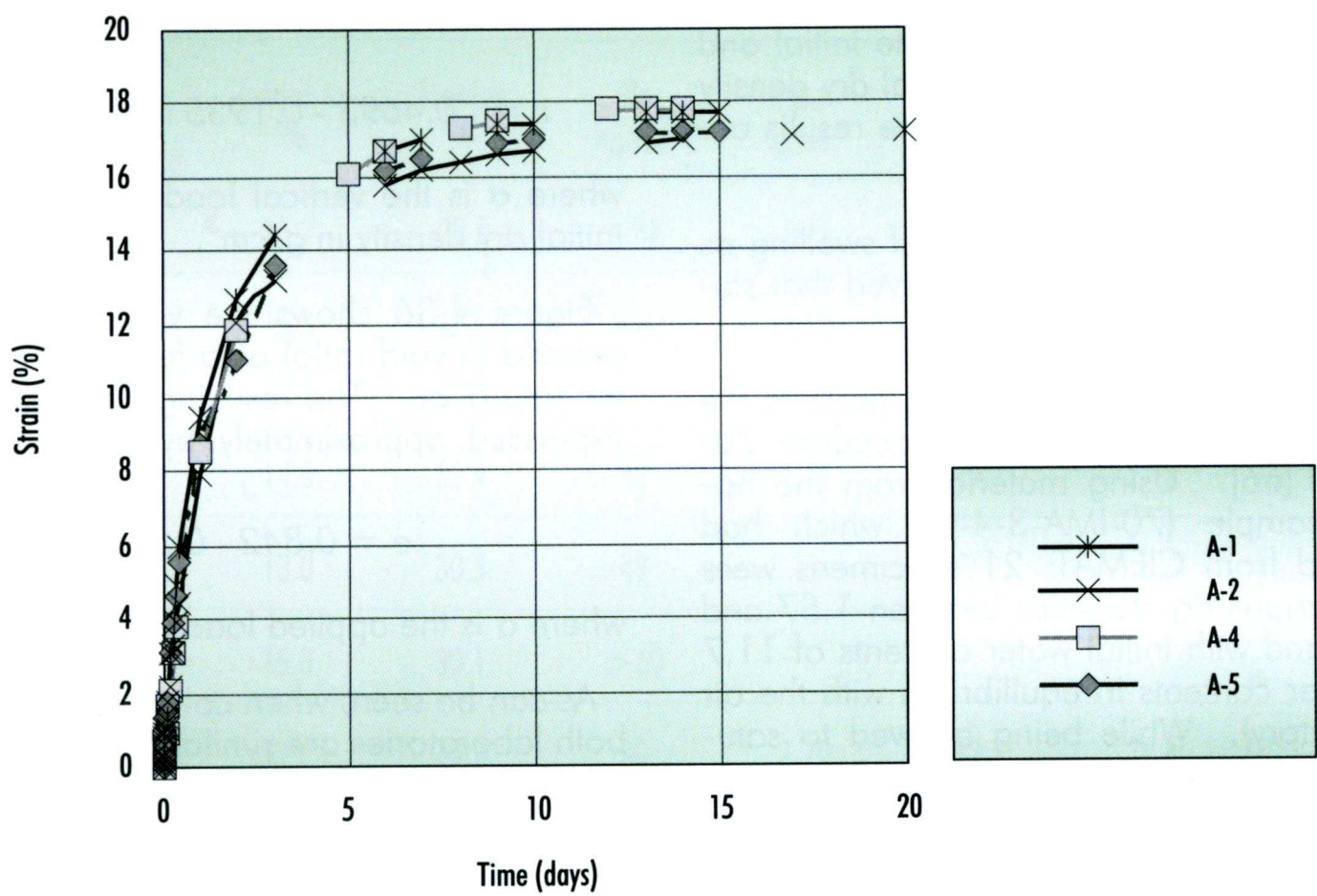


Figure 4.27: Swelling vs. time under a vertical load of 0.5 MPa (70-IMA-3-4-0, A samples).

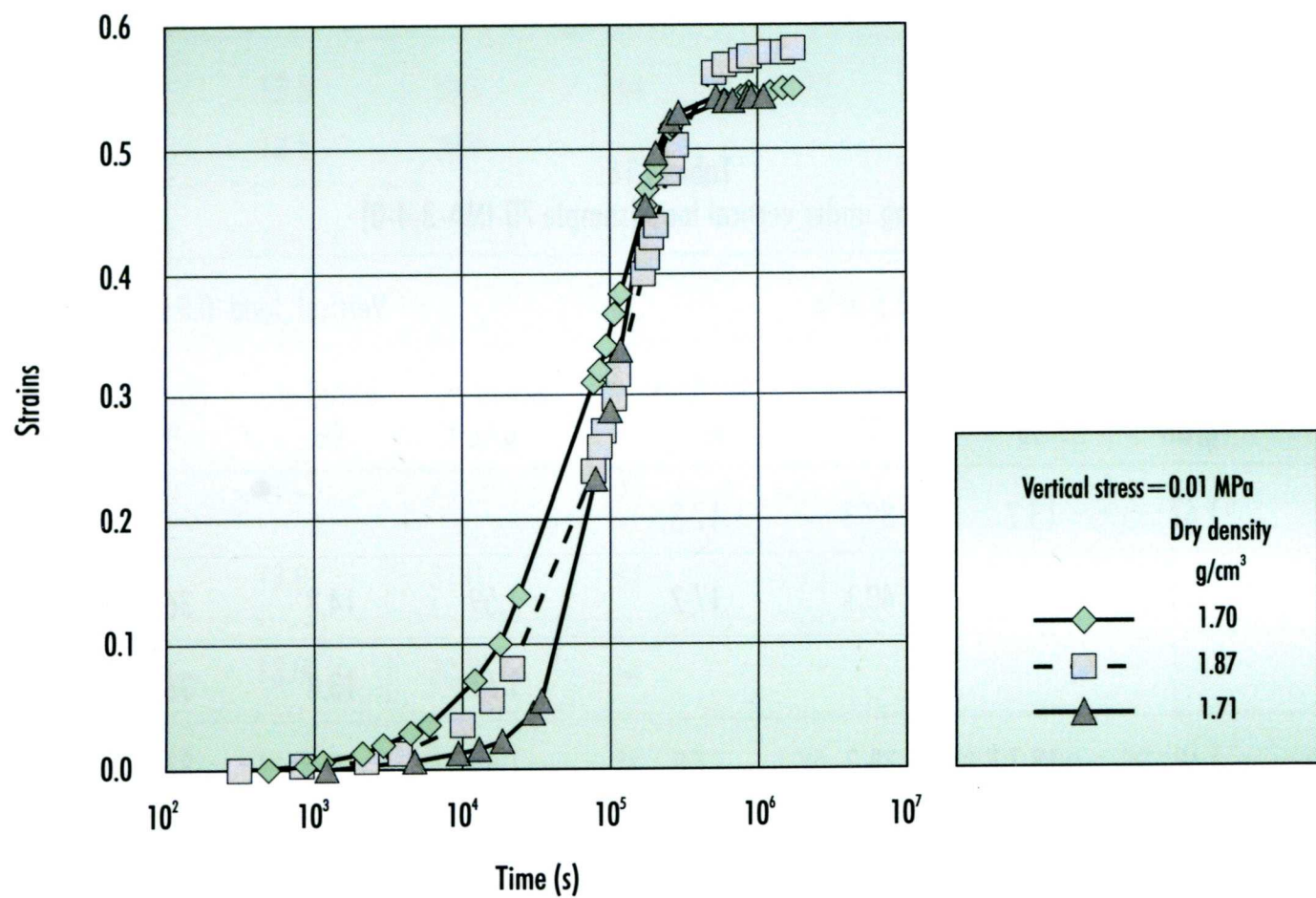


Figure 4.28: Evolution with time of swelling strains during saturating tests under load. Vertical stress = 0.01 MPa.

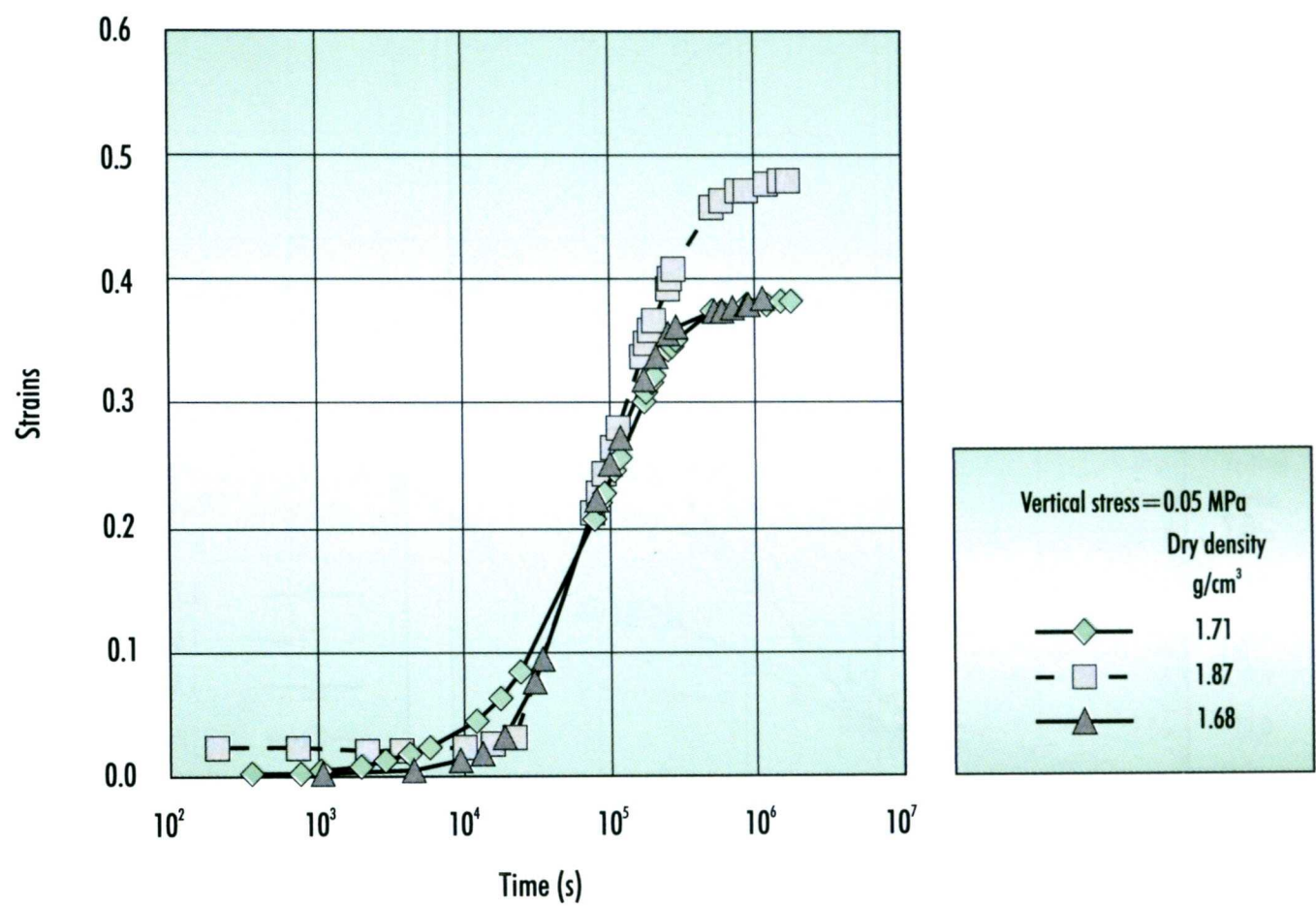


Figure 4.29: Evolution with time of swelling strains during saturating tests under load. Vertical stress = 0.05 MPa.

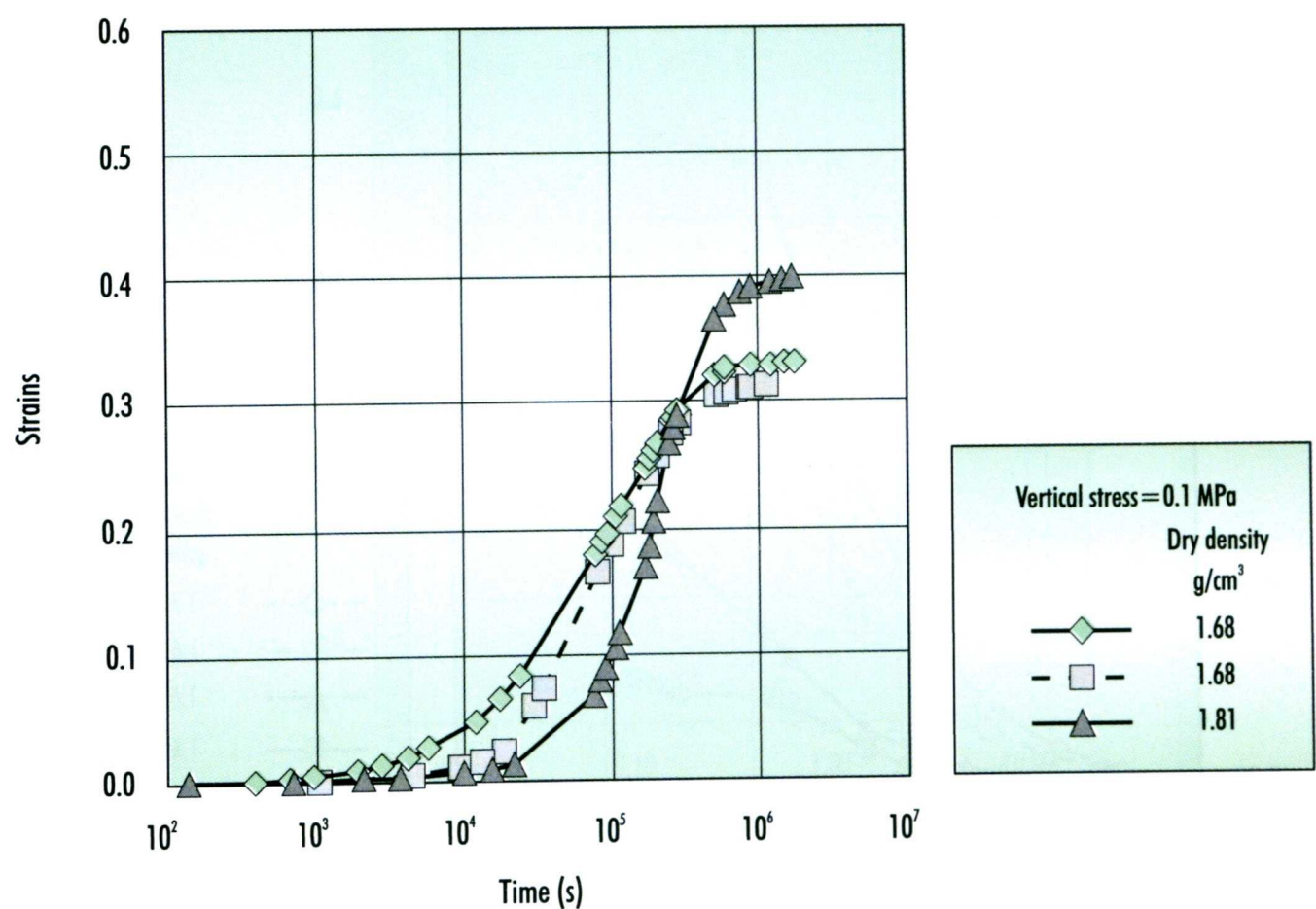


Figure 4.30: Evolution with time of swelling strains during saturating tests under load. Vertical stress = 0.1 MPa.

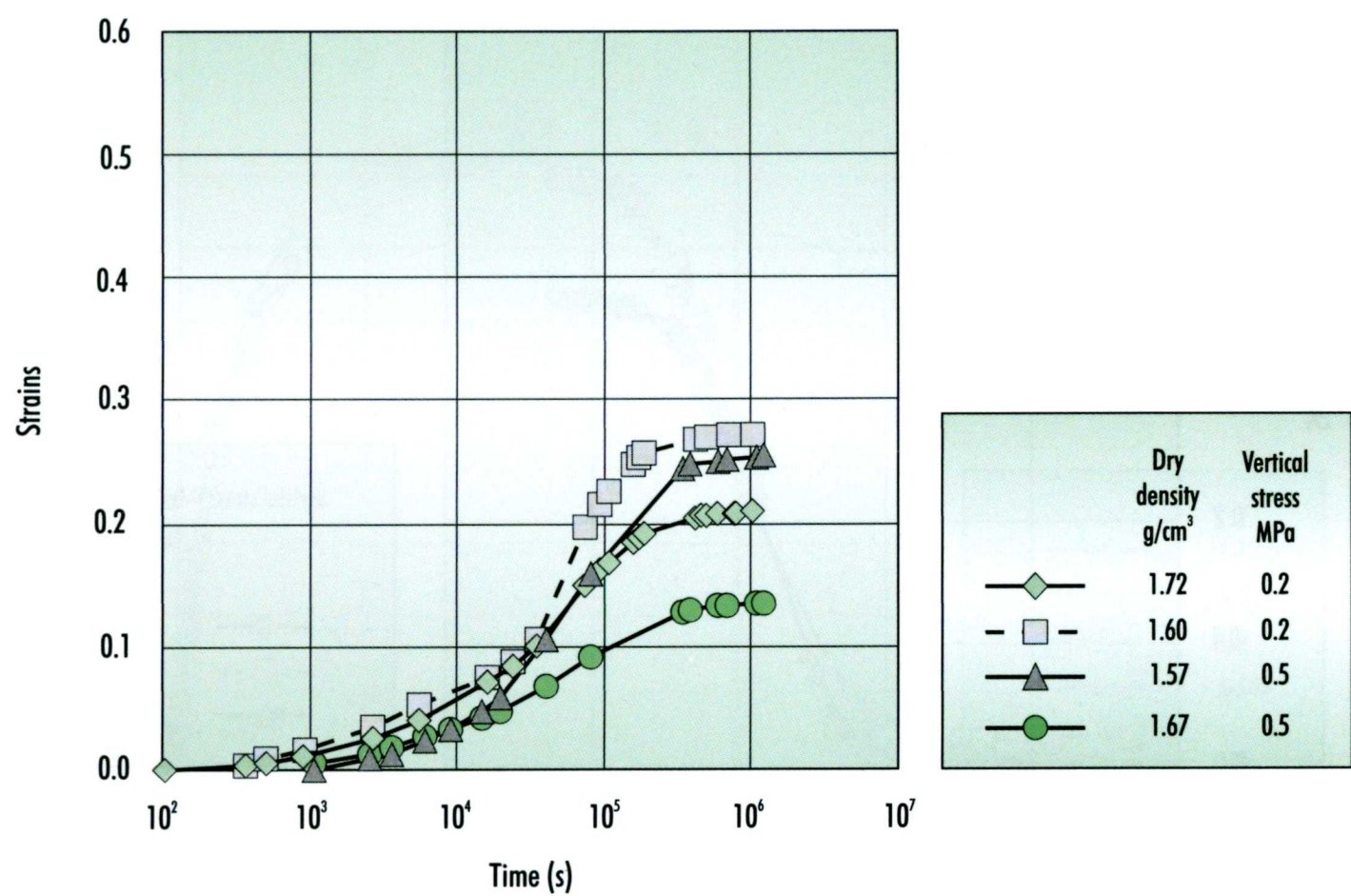


Figure 4.31: Evolution with time of swelling strains during saturating tests under load. Vertical stress = 0.2; 0.5 MPa.

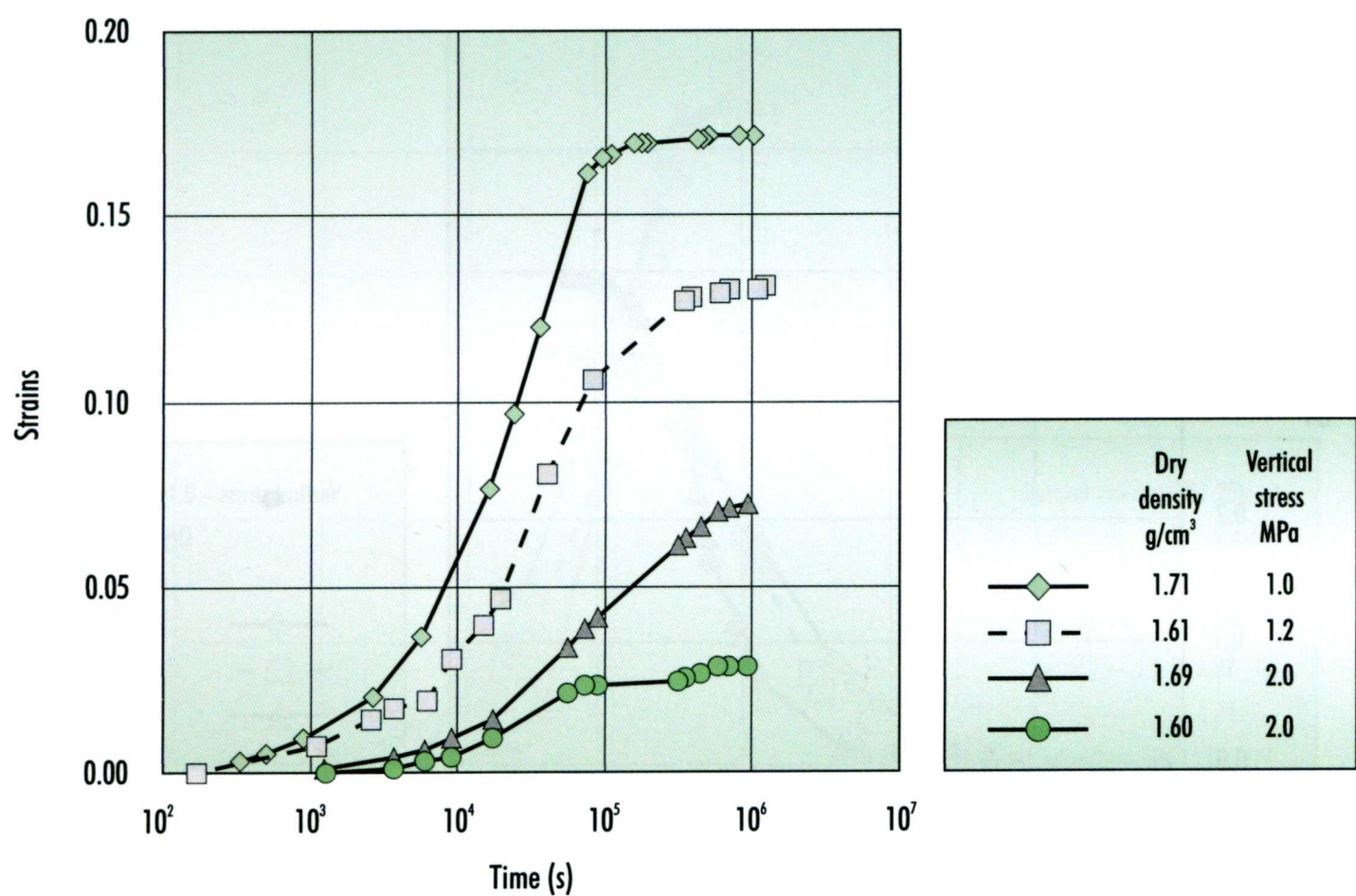


Figure 4.32: Evolution with time of swelling strains during saturating tests under load. Vertical stress = 1; 1.2; 2.0 MPa.

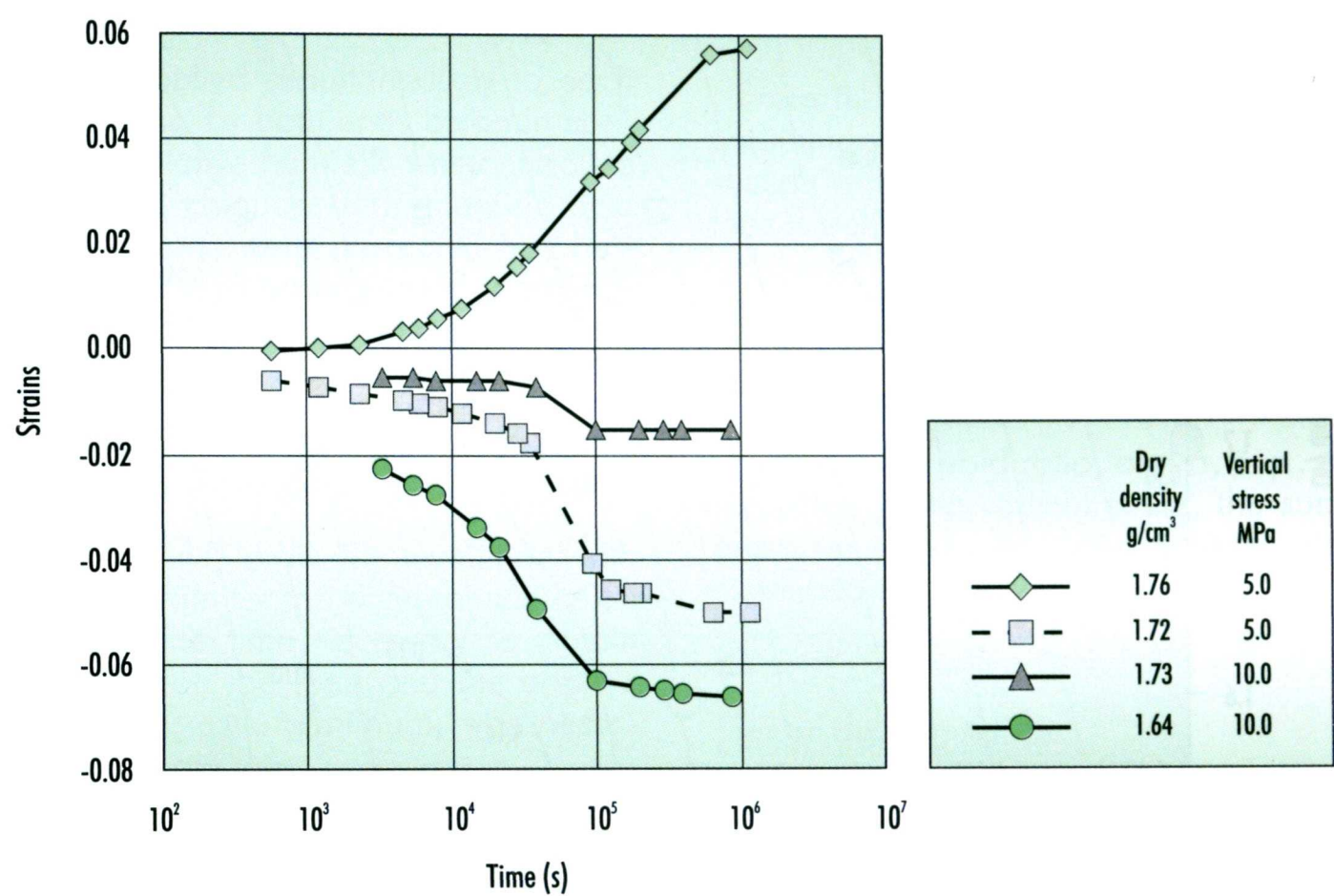


Figure 4.33: Evolution with time of swelling strains during saturating tests under load. Vertical stress = 5; 10 MPa.

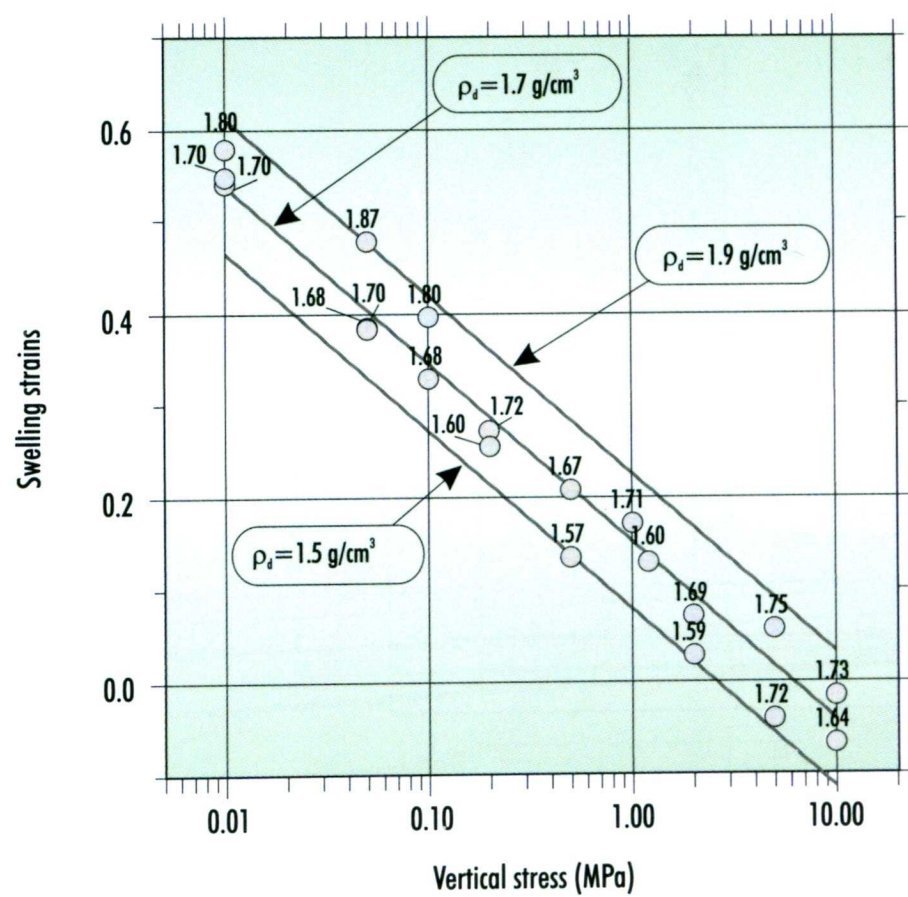


Figure 4.34: Swelling strains for applied vertical stresses. The initial dry density is shown near each point.

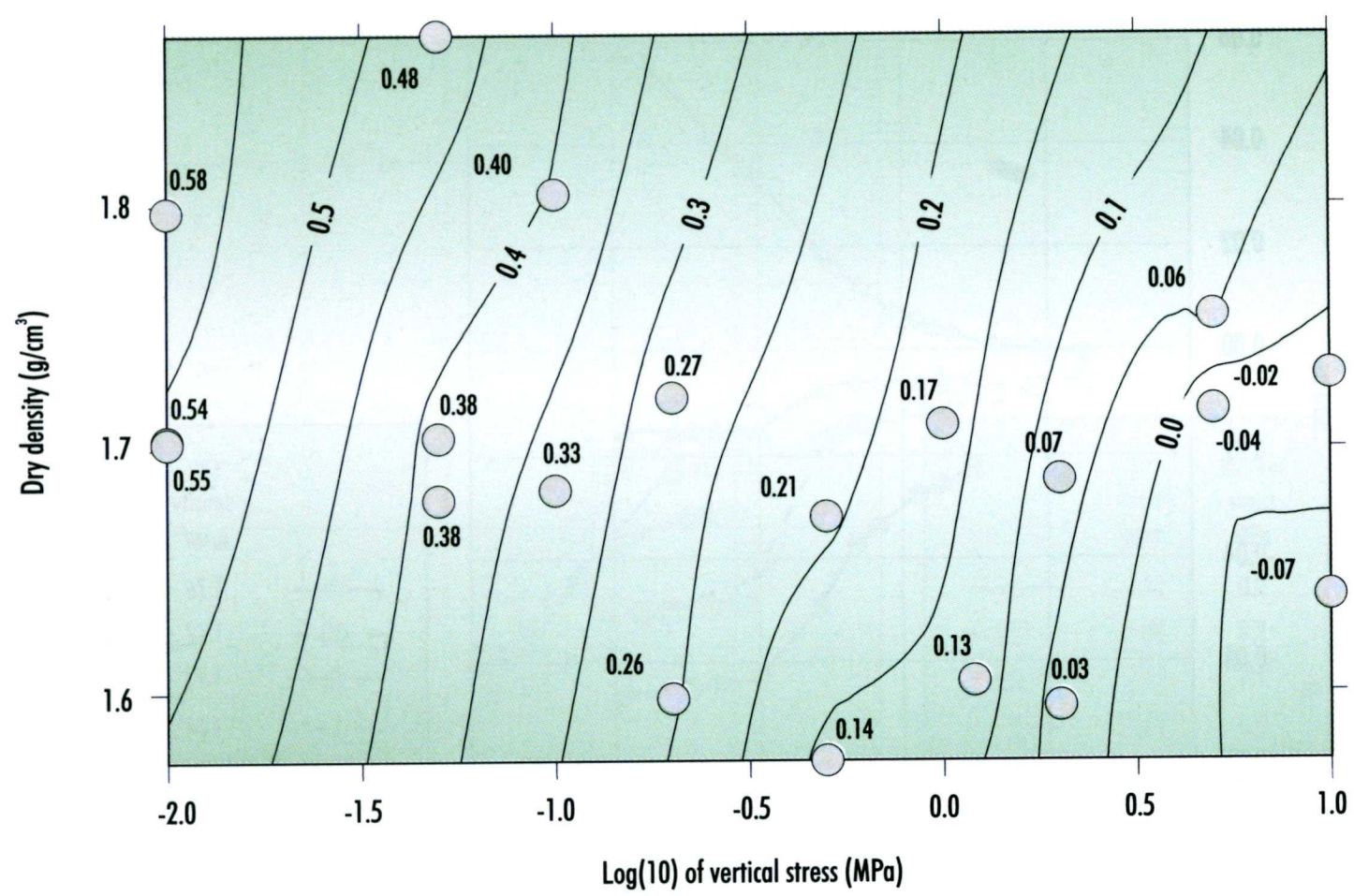


Figure 4.35: Swelling strains for different applied vertical loads and initial dry densities.

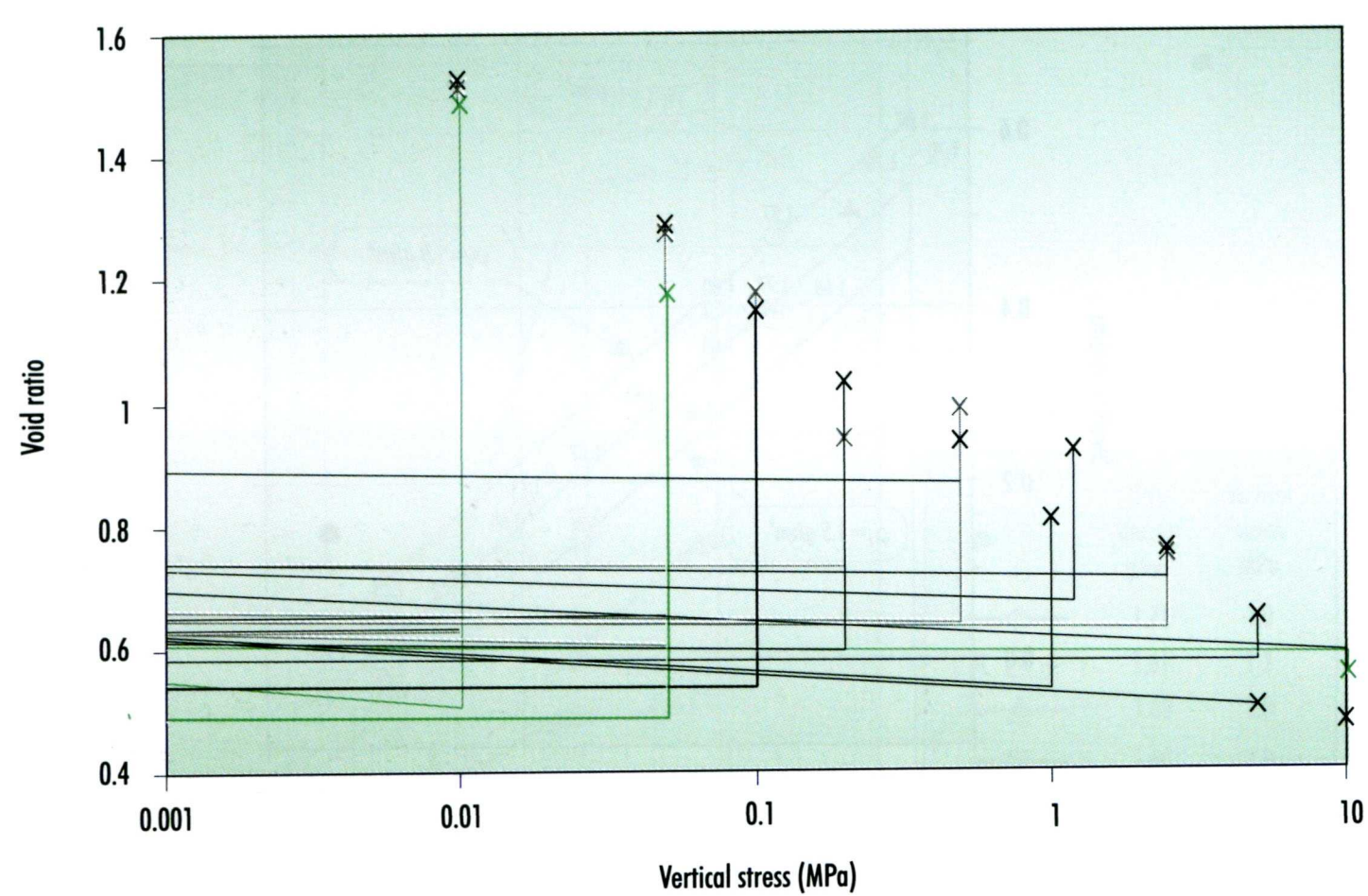


Figure 4.36: Final void ratio for wetting under load tests.

followed by suctions, in MPa, of 97, 360, 135, and 0 (total saturation).

The suction was applied simultaneously to each of the three specimens, by both ends being in contact with air whose relative humidity was controlled by means of sodium chloride and sulphuric acid solutions. The solutions were placed in two hermetic containers and were transferred to the specimens, from the containers, by a constant air flow provided by a small pump.

The methodology and devices are described in Procedure 70-UPC-G-0-04 [67].

Figures 4.37 to 4.41 show the evolution with time of the vertical strain for the different values on the suction path. The time necessary to obtain equilibrium depends on the load and the applied suction, but a month, as a minimum, was necessary to reach small strain rates.

Figures 4.42 to 4.44 show the void ratio versus the complete suction path for each vertical load applied.

Figure 4.45 shows the void ratio as a function of the vertical load for each suction. The same figure

also shows the final void ratio for the swelling under load tests.

From the results of these tests can be deduced a preliminary state surface for the case of decreasing suction with small changes of stress. The state surface is defined by the following equation

$$e = 0.8169 - 0.1268 \log (\sigma - p_a) - 0.04968 \log (s + p_{atm}) + 0.05409 \log (\sigma - p_a) \log (s + p_{atm})$$

where e is the void ratio, and σ , s , p_a and p_{atm} are, respectively, the vertical stress, the suction, the air pressure, and the atmospheric pressure, all expressed in MPa.

Elastic shear modulus

These tests were made by UPC-DIT, following Procedure 70-UPC-G-0-06 [68]. Ten specimens, compacted to a dry density between 1.53 and 1.73 g/cm³ and with degrees of saturation of 0.15, 0.50, and 0.90, were tested in a resonant column to obtain the elastic shear modulus (G) for

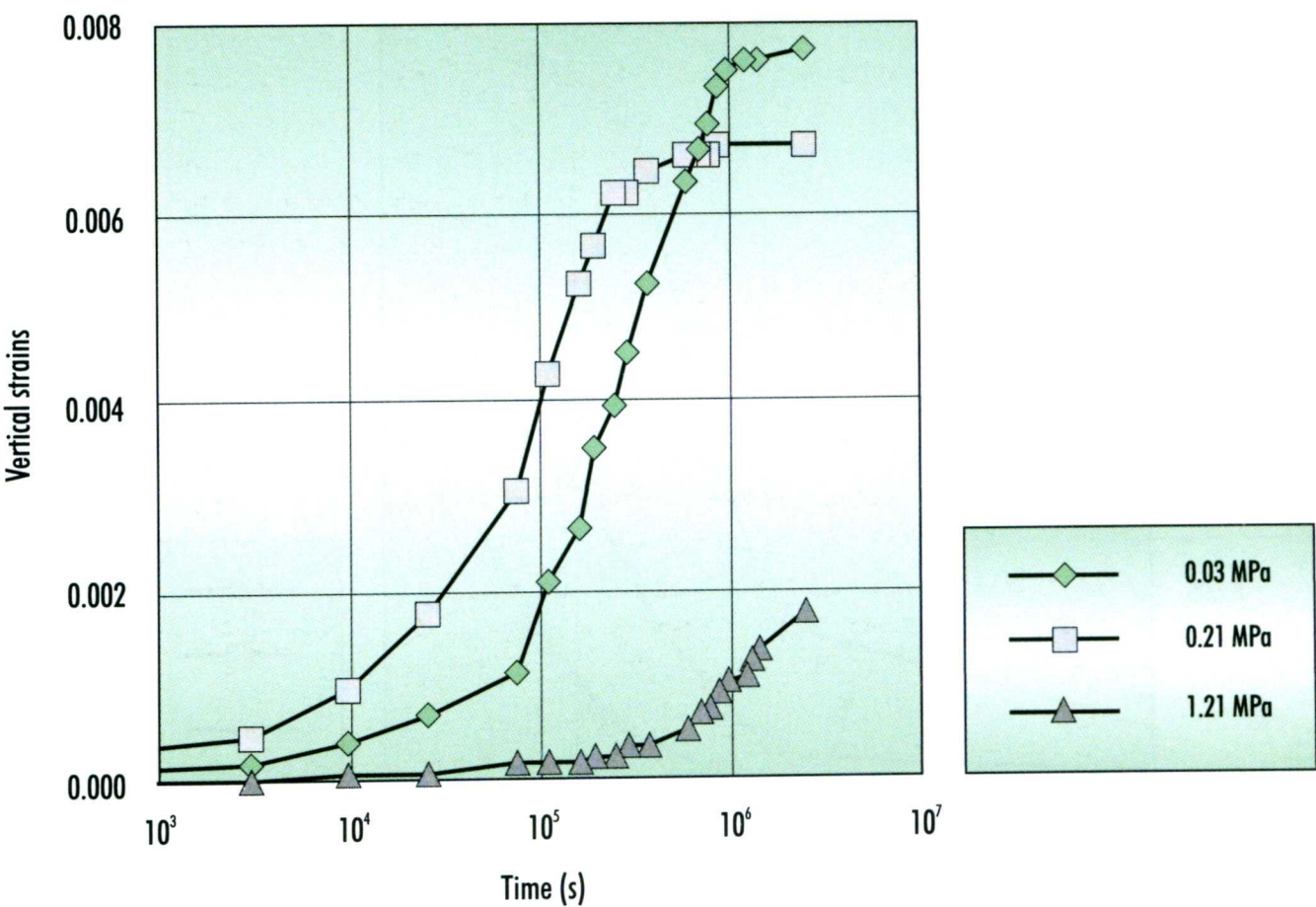


Figure 4.37: Evolution with time of the strains for change of suction . From 130 MPa (initial suction) to 97 MPa.

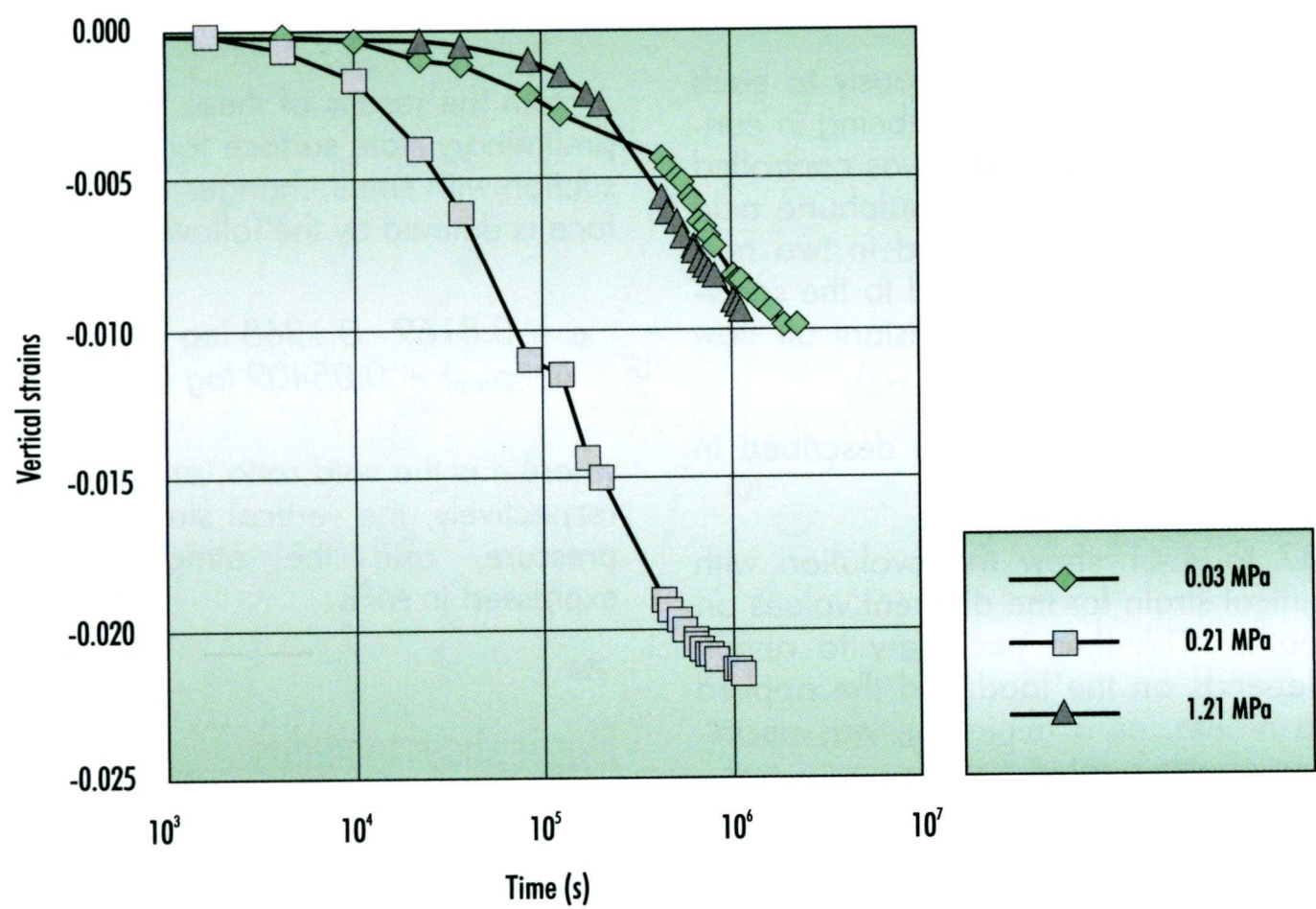


Figure 4.38: Evolution with time of the strains for change of suction. From 97 MPa to 360 MPa.

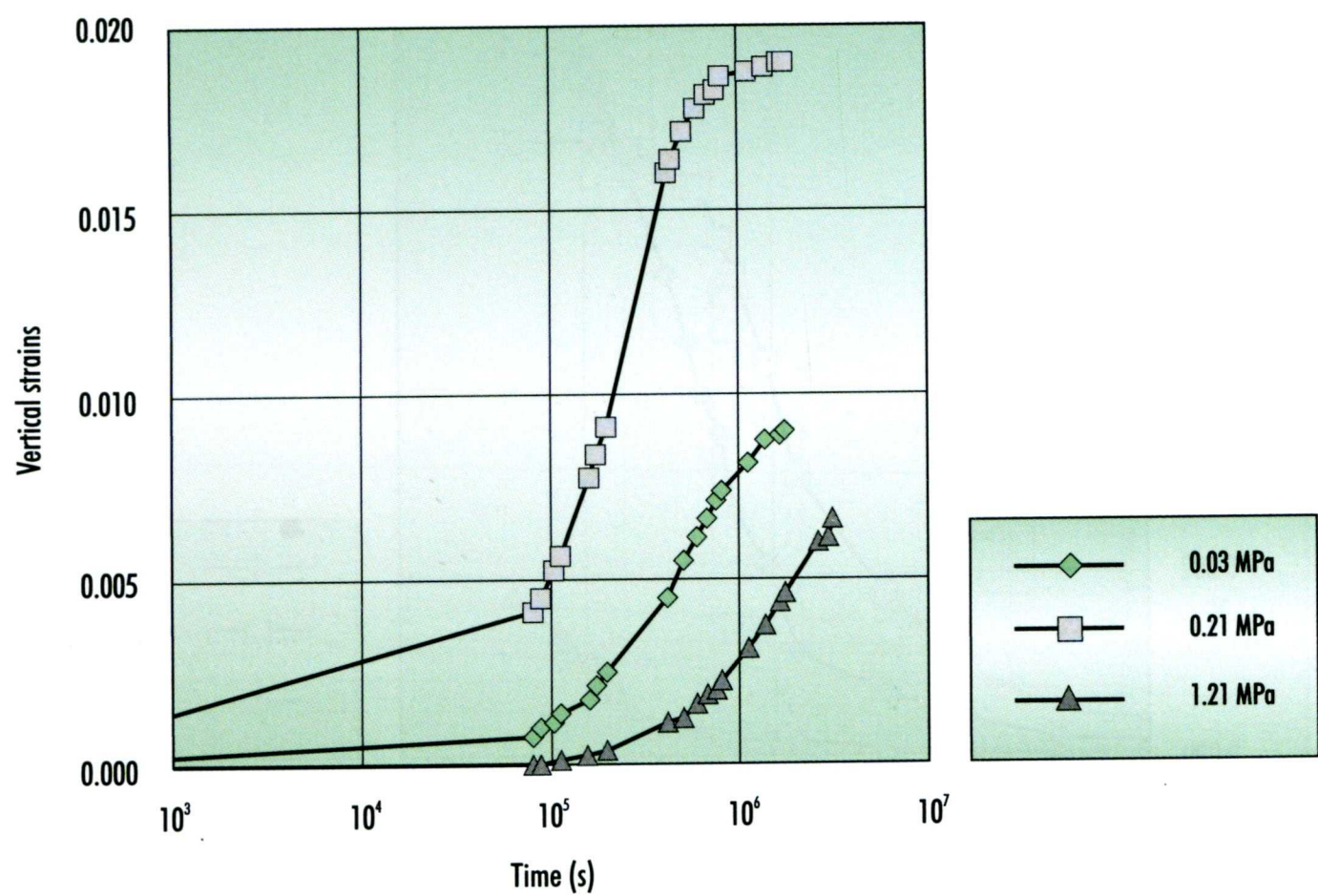


Figure 4.39: Evolution with time of the strains for change of suction. From 360 MPa to 135 MPa.

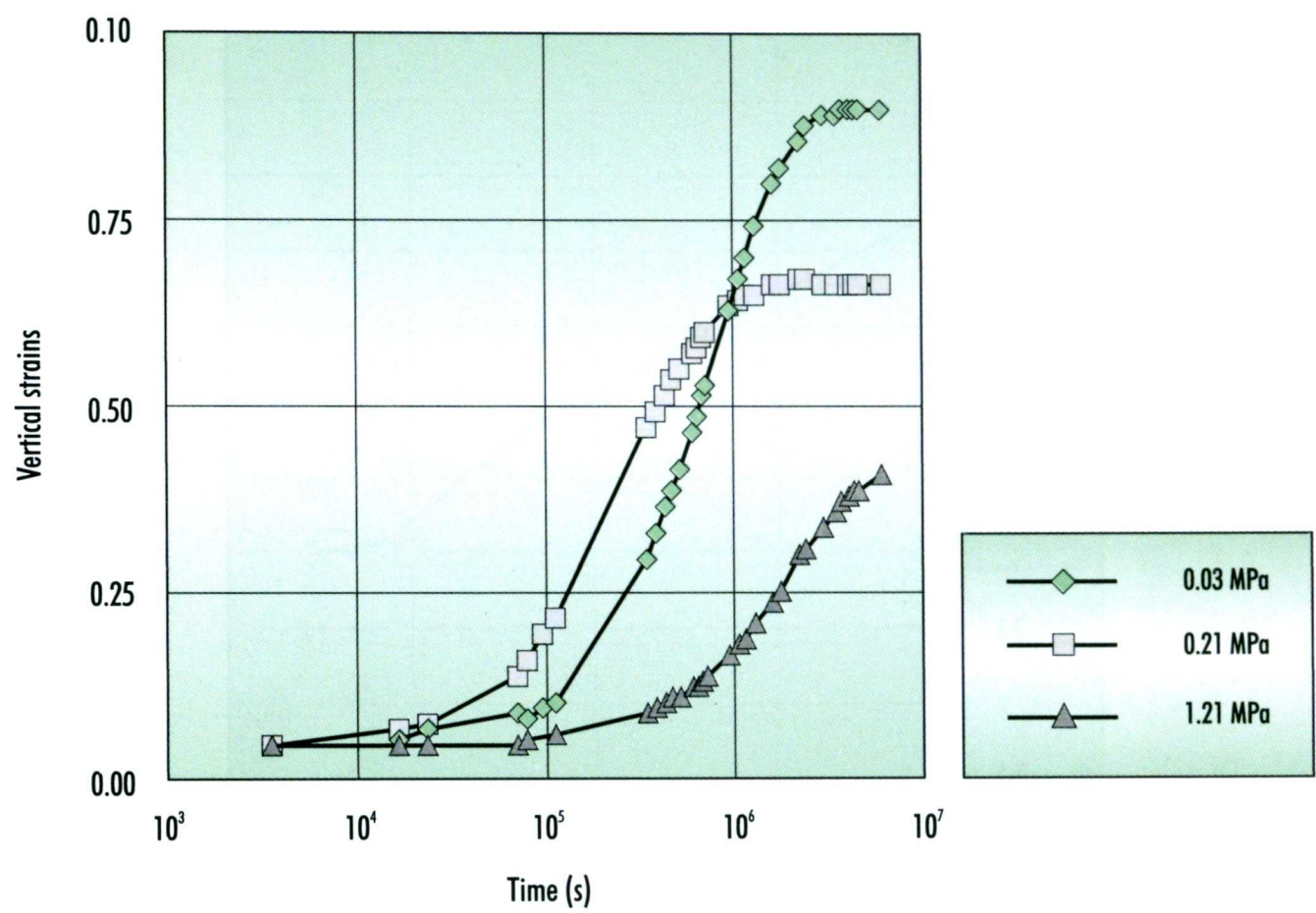


Figure 4.40: Evolution with time of the strains for change of suction. From 135 MPa to 22 MPa.

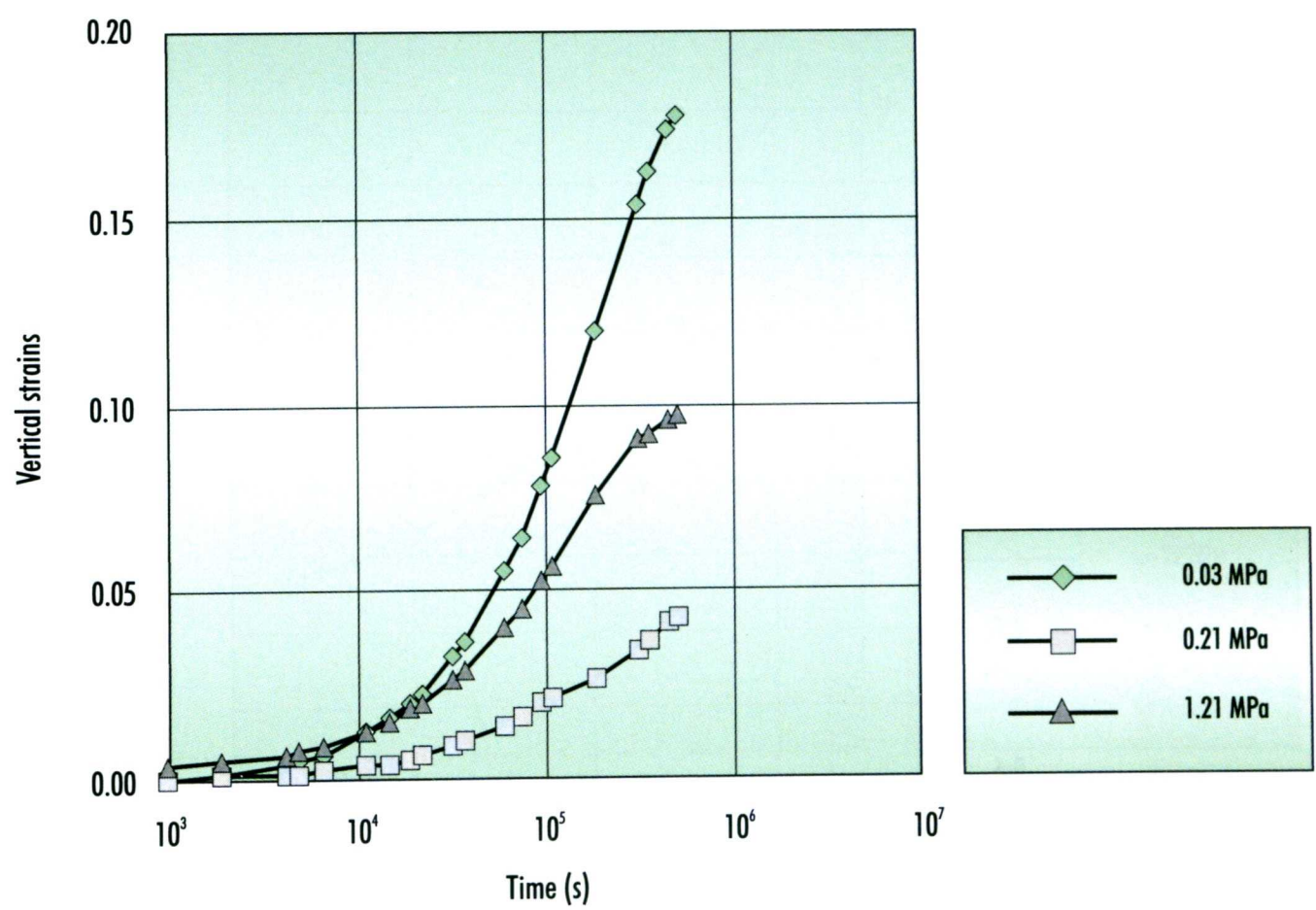


Figure 4.41: Evolution with time of the strains for change of suction. From 22 MPa to 0 MPa.

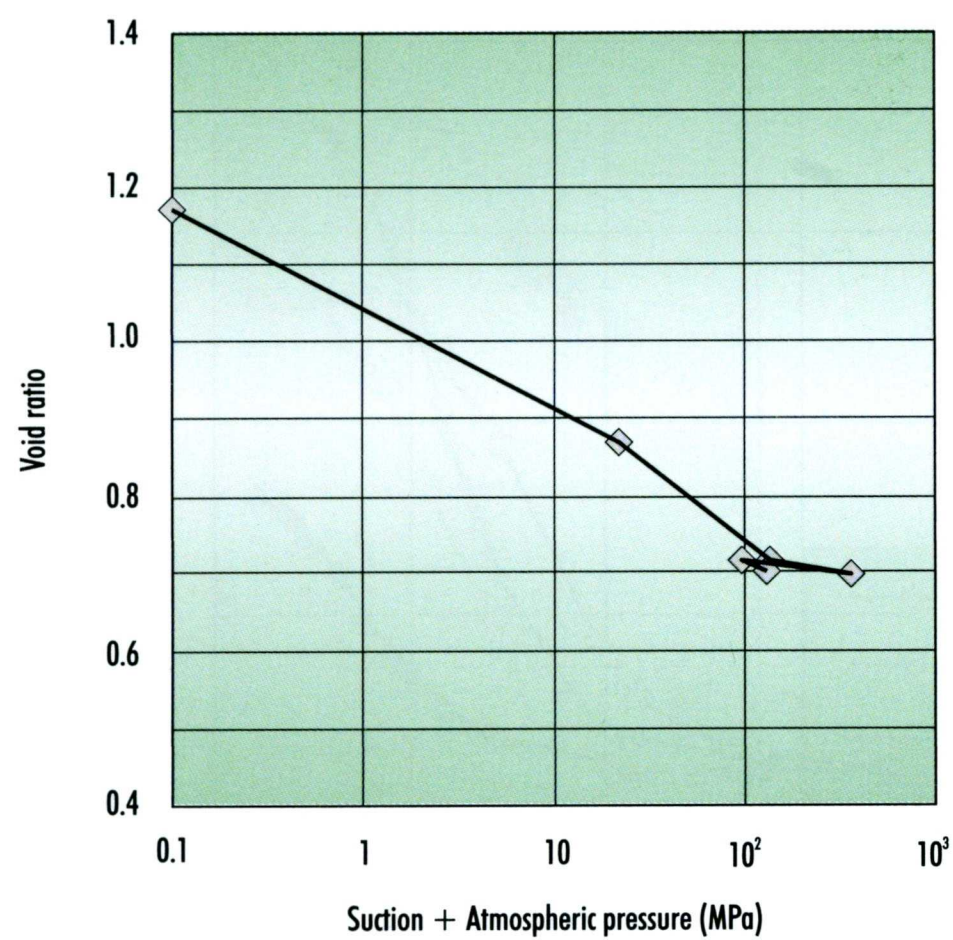


Figure 4.42: Suction / void ratio in oedometer tests. Vertical stress 0.03 MPa.

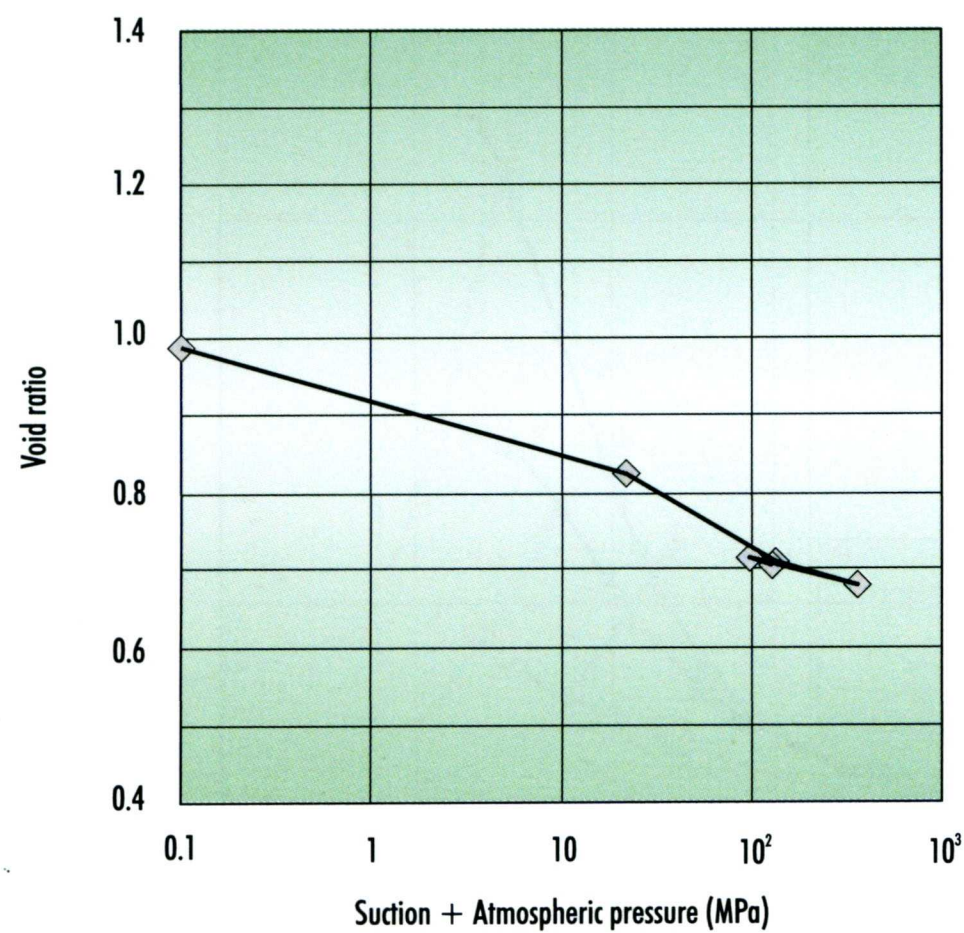


Figure 4.43: Suction / void ratio in oedometer tests. Vertical stress 0.21 MPa.

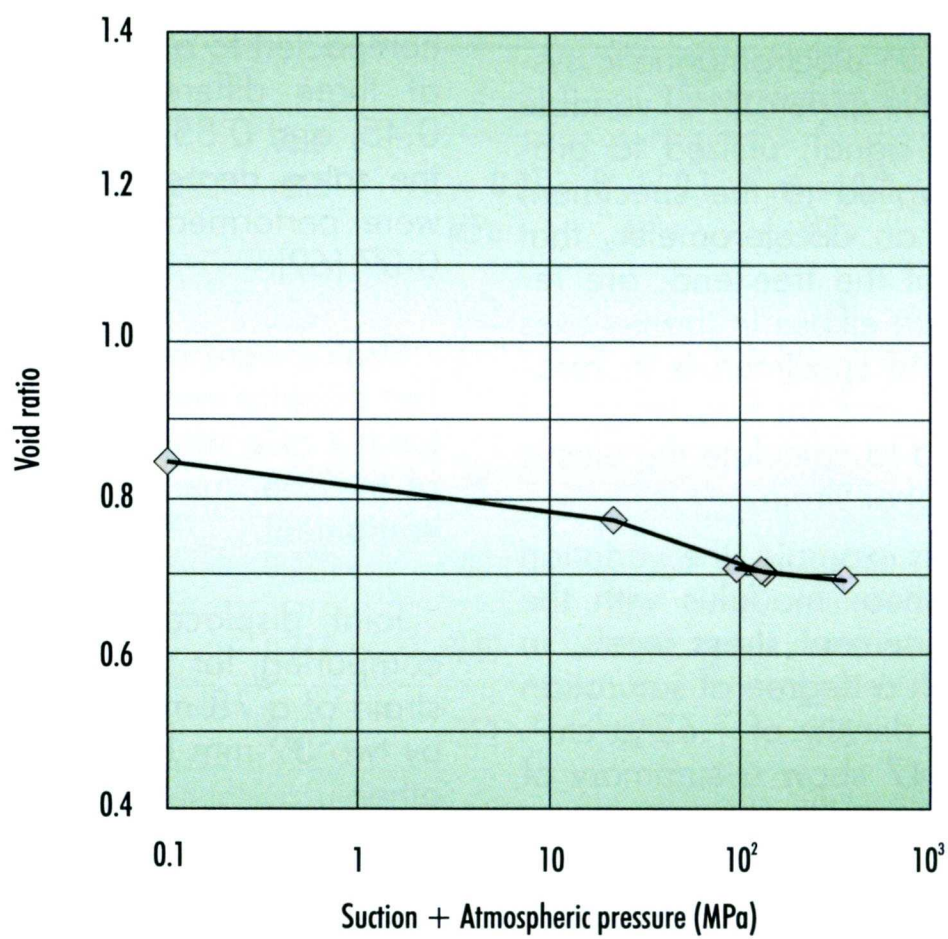


Figure 4.44 : Suction / void ratio in oedometer tests. Vertical stress 1.21 MPa.

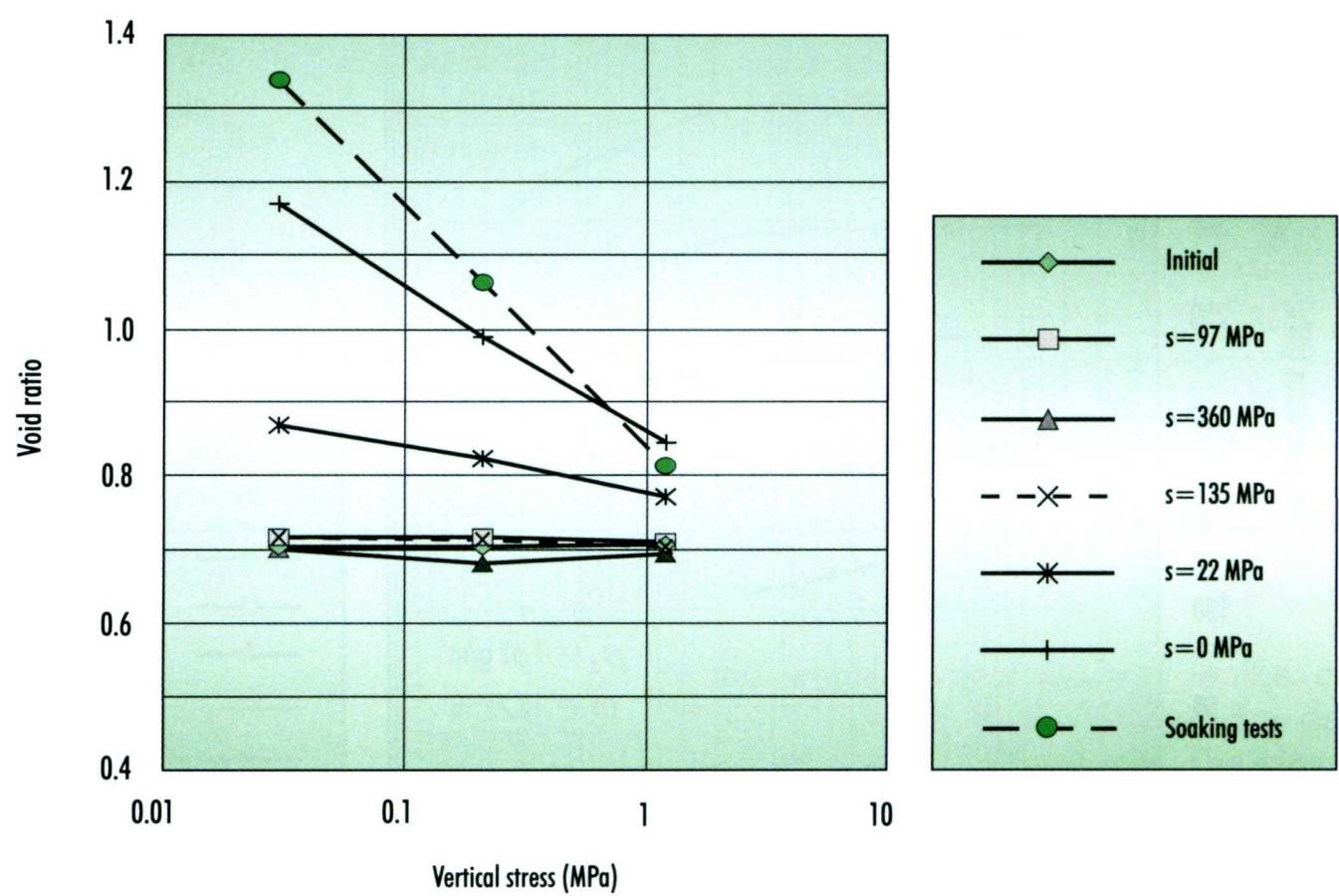


Figure 4.45: Results of all wetting tests showing the void ratio variation with applied vertical loads for different values of equilibrium suction. Also are indicated the results of the saturation tests in conventional oedometer.

small shear deformations ($10^{-6} \leq \gamma \leq 10^{-4}$). A fixed free-type resonant column device was utilized.

The device is based on an electromagnetic system that is fed by a sine wave generator of variable frequency. The electrical signal, utilized to produce the cyclic torque applied to the specimen, and the output signal of an accelerometer, that measures the movement of the free end, are recorded and they produce an ellipse in the x-y axes of an oscilloscope when the specimen is in resonance. The values of the resonant frequency and the acceleration are utilized to calculate the elastic shear modulus and the level of strain.

Figure 4.46 shows, as an example, the variation in the measured elastic shear modulus with the shear strain and the confinement stress levels, in the case of a specimen with a degree of saturation of 0.50 and an initial dry density of 1.62 g/cm^3 . Table 4.17 and Figure 4.47 show a summary of the results obtained.

Joint stiffness

UPC-DIT made four unconfined compression tests on cylindrical (38-mm diameter) compacted specimens in order to obtain, in the joints between

the bentonite blocks, the relationship of normal stress with displacement. The specimens were compacted to a nominal dry density of 1.63 g/cm^3 at three different degrees of saturation (0.15, 0.45, and 0.85 with two specimens compacted to the same degree of saturation, 0.45.) The tests were performed following Procedure 70-UPC-G-0-07 [69].

Strains were measured by means of two Hall effect displacement transducers, parallel to the longitudinal axis, mounted directly on the lateral surface of the specimens to avoid bedding effects of the equipment.

Joint displacements have been evaluated by comparing, for the same stress level, the measured strain of a 78-mm high specimen and one formed by two 39-mm high specimens, one on top of the other.

Figures 4.48 to 4.50 show the relationship of stress to measured strain in the specimen (with and without joints) and the normal stress as a function of the displacement of the joint. The unconfined compressive strength and Young's modulus that were obtained are shown in Table 4.18.

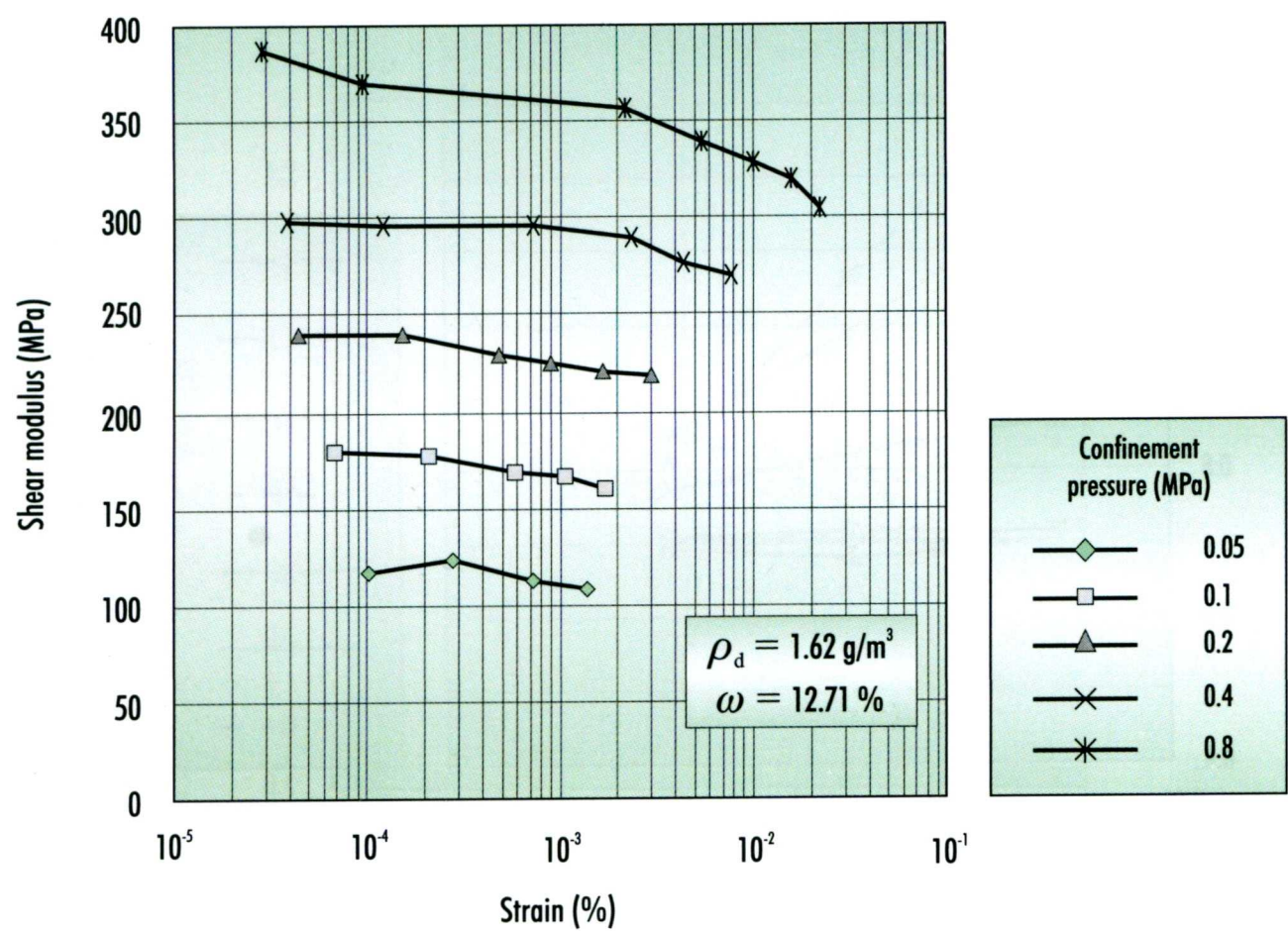


Figure 4.46: Shear modulus for various confining loads and shear strains.

Table 4.17
Shear elastic modulus from resonant column tests

Dry density g/cm ³	Water content	Degree of saturation	Void ratio	Shear modulus G (MPa)				
				$\sigma_3 = 0.01$ MPa	$\sigma_3 = 0.1$ MPa	$\sigma_3 = 0.2$ MPa	$\sigma_3 = 0.4$ MPa	$\sigma_3 = 0.8$ MPa
1.58	14.7	54	0.757	140	207	245	300	370
1.66	13.6	57	0.668	211	223	270		
1.54	3.3	12	0.802	78	106	174	208	326
1.56	2.8	10	0.777	89	106	138		
1.62	24.6	95	0.717	240	270		331	336
1.66	21.2	87	0.677	252	296	370	429	502
1.65	4.7	19	0.685	74	90	137	190	310
1.72	10.4	47	0.615	200	219	293	381	429
1.68	3.7	16	0.652	61	89	141	200	290
1.62	12.7	50	0.713	122	180	240	299	387

Table 4.18
Strength and stiffness of bentonite specimens compacted at a dry density of 1.63 g/cm³

Degree of saturation	Compressive strength (MPa) With joint	Compressive strength (MPa) Without joint	Young modulus (MPa), $\varepsilon = 0.5\%$ Without joint
0.15	0.82	0.49	78
0.45	2.19	2.18	377
0.45	2.68	2.23	436
0.85	4.43	4.76	601

4.4.2. Hydraulic tests

Saturated hydraulic conductivity

Following Procedure 70-IMA-G-0-14 [70], 6 un-mixed samples (F samples) have been tested to determine the saturated hydraulic conductivity. From these samples, 12 specimens were compacted at

the water content existing in the closed storage bags, 6 each to a nominal dry density of 1.60 g/cm³ and of 1.70 g/cm³. The clay was uniaxially compacted in cylindrical rings of stainless steel. The specimens, 5.0 cm in diameter and 2.5 cm in height, were placed between porous stones. Later, water was injected at the top and bottom under a pressure of 0.6 MPa. Once the specimens were saturated, a hydraulic gradient was applied: the

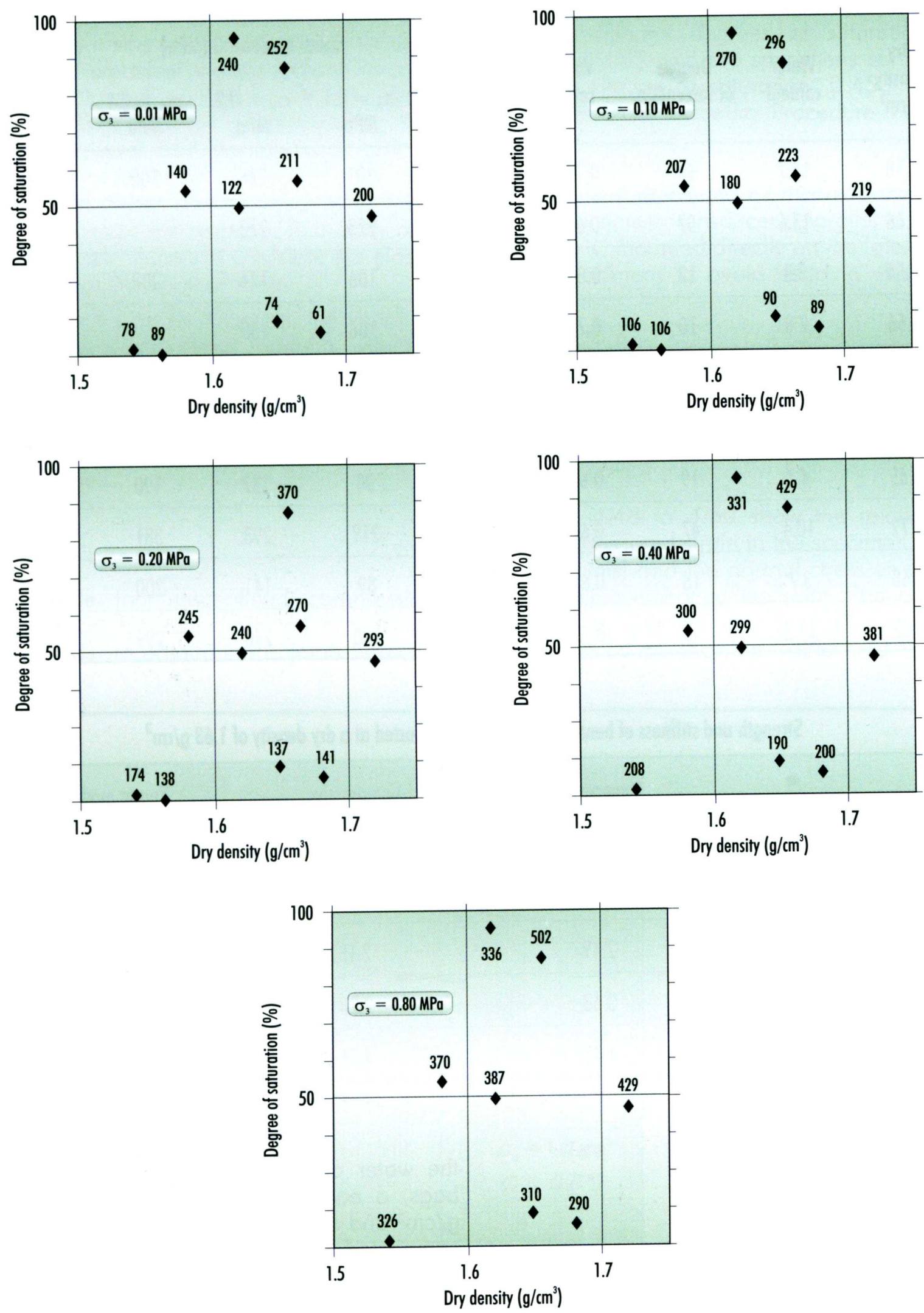


Figure 4.47: Shear modulus at small shear strains for various degrees of saturation and dry densities.

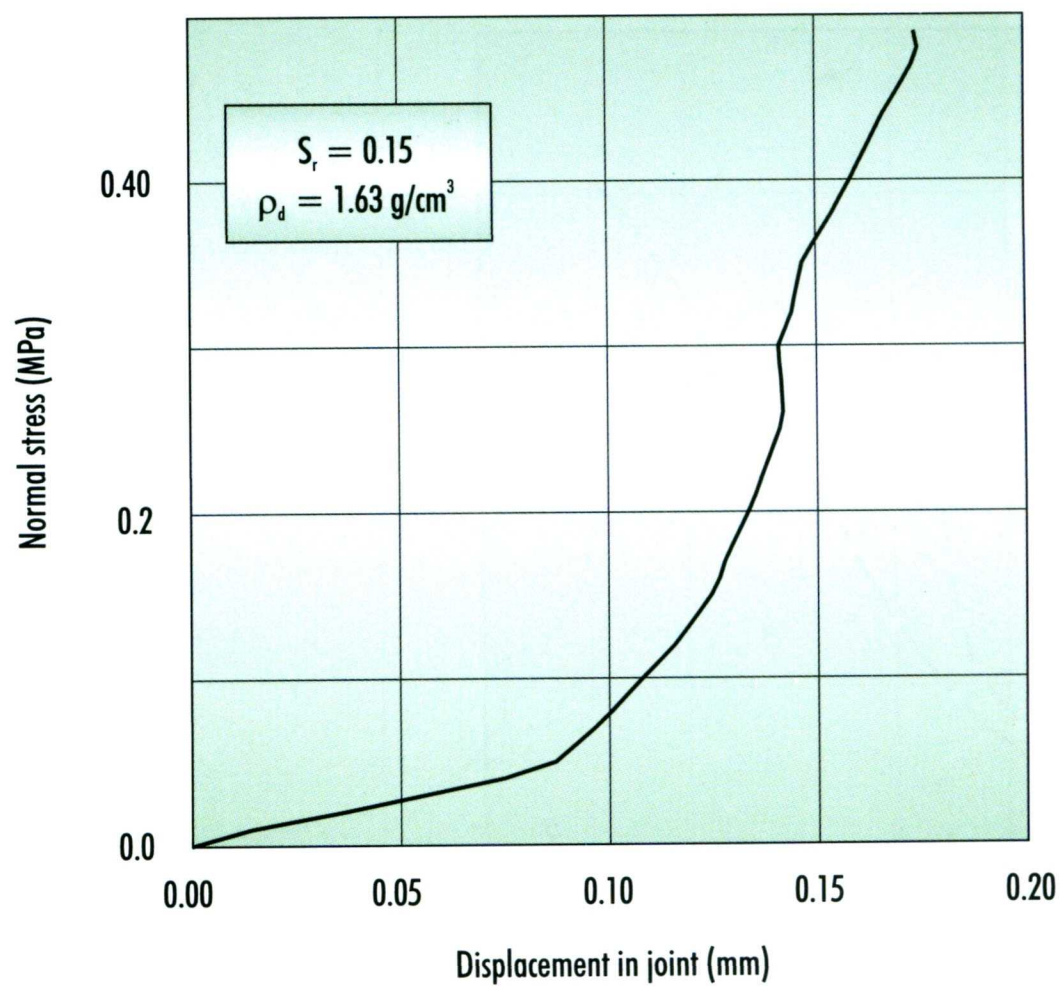
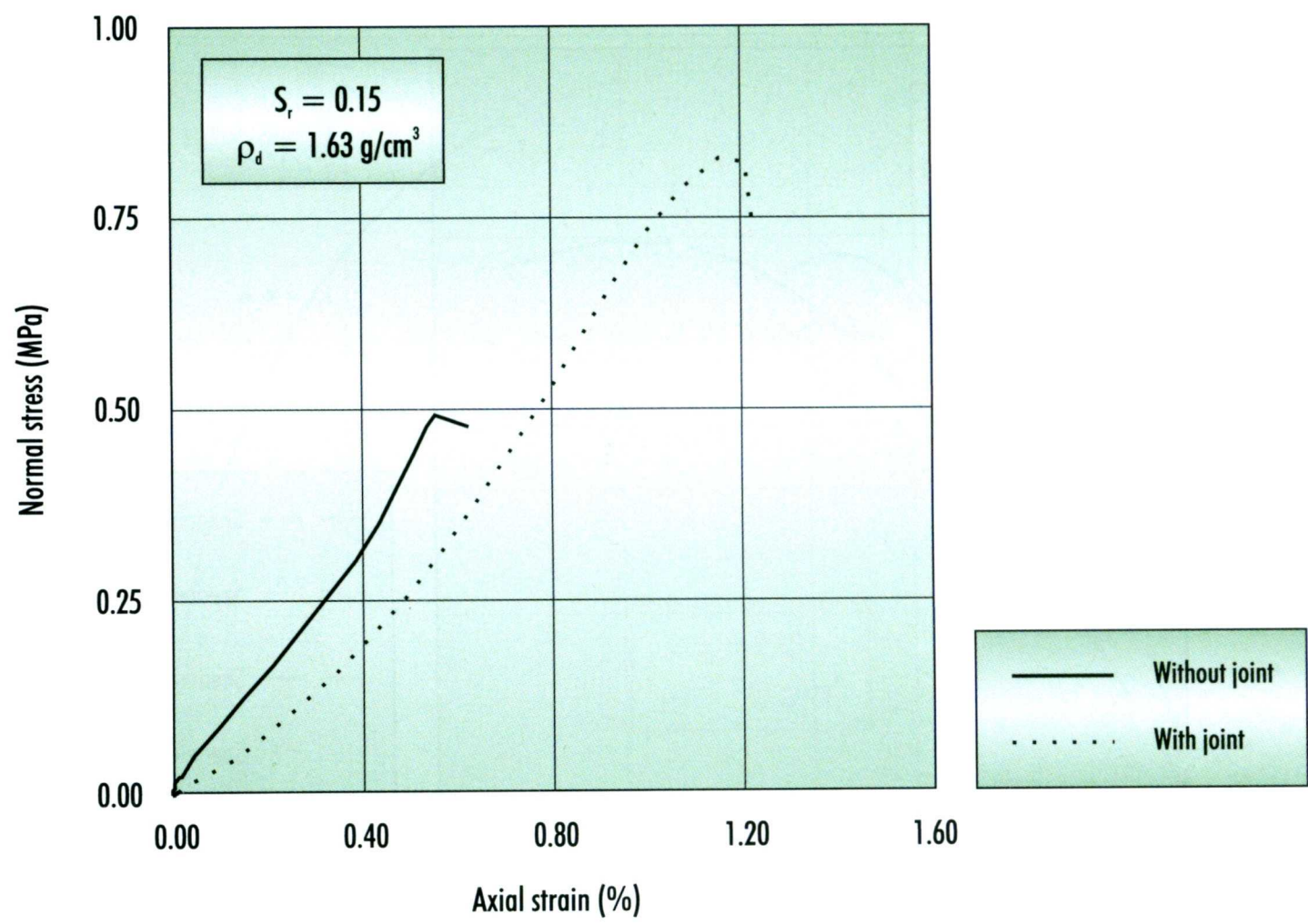


Figure 4.48: Normal stress vs. displacement in compacted bentonite sample at a degree of saturation of 0.15.

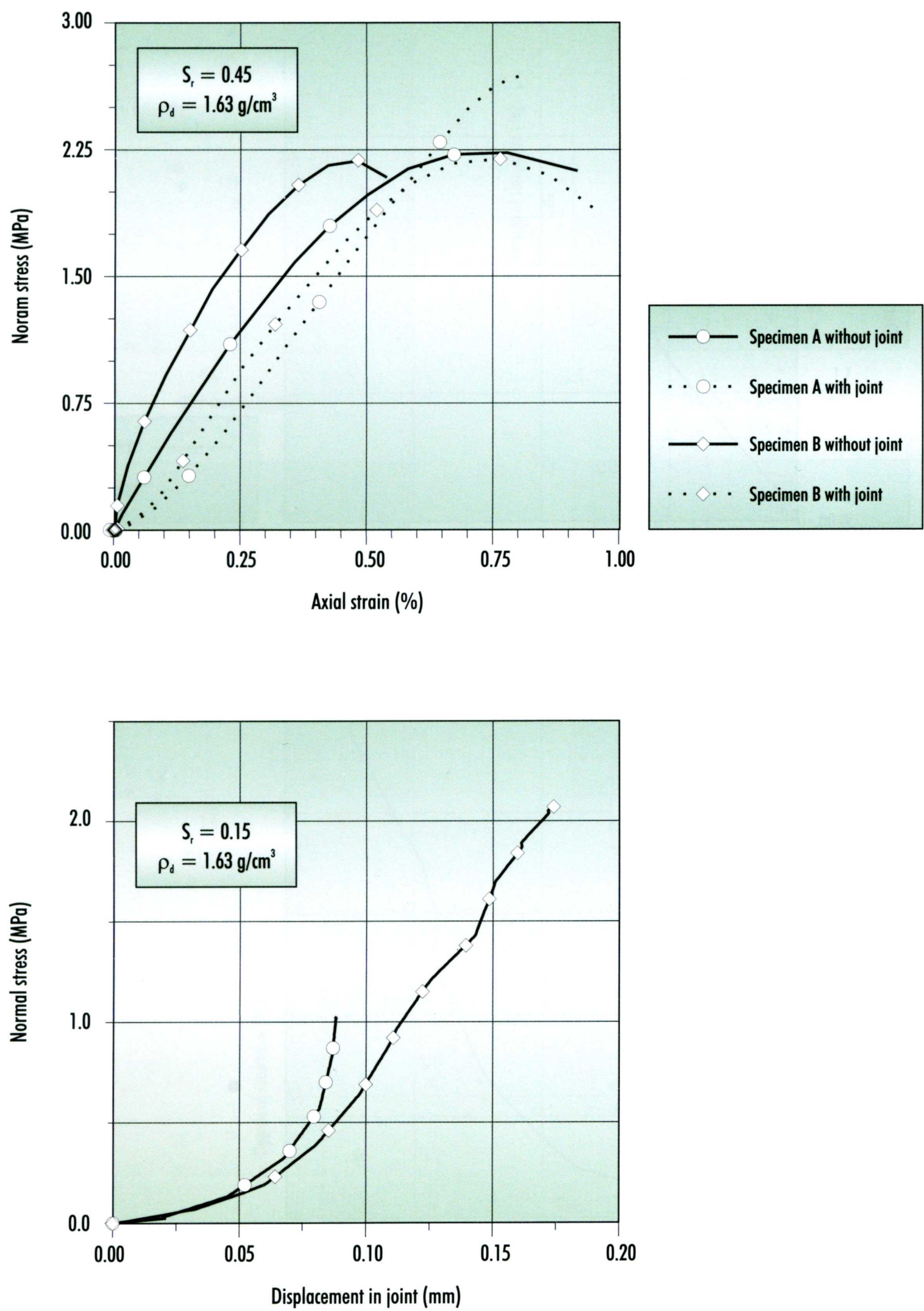


Figure 4.49: Normal stress vs. displacement in compacted bentonite sample at a degree of saturation of 0.45.

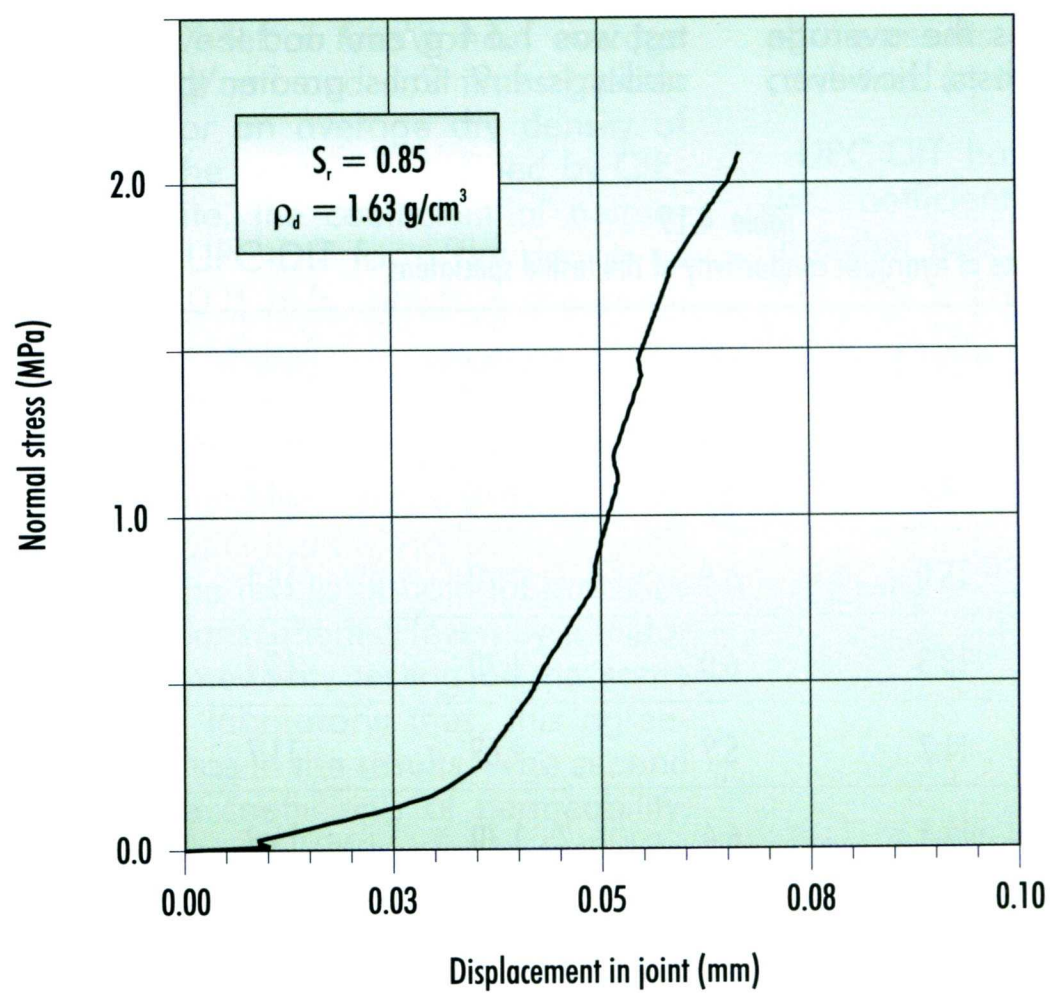
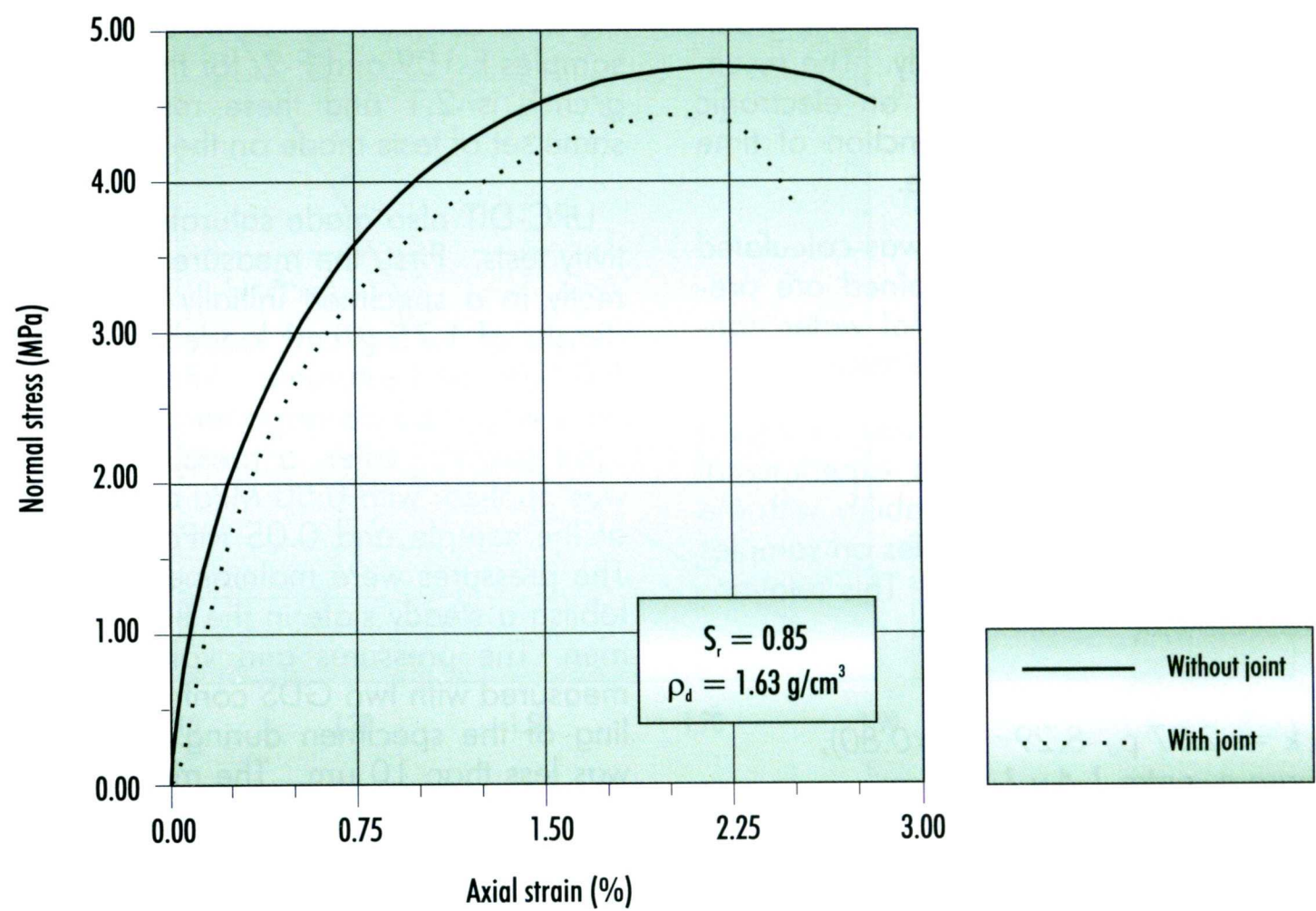


Figure 4.50: Normal stress vs. displacement in compacted bentonite sample at a degree of saturation of 0.85.

pressure was raised to 5.2 MPa at the bottom, for dry densities of 1.60 g/cm³, and to 7.2 MPa, for dry densities of 1.70 g/cm³, giving hydraulic gradients of 18400 or 25600, respectively. The resulting water flow was measured, with an electronic volume change apparatus, as a function of time until the flow reached the steady state.

The coefficient of permeability, *k*, was calculated by Darcy’s law and the values obtained are presented in Table 4.19, where the final water contents and final dry densities are also shown.

The values obtained are represented in Figure 4.51. This figure also includes an experimental graphical relationship of the permeability with the dry density obtained in the prior studies on samples from the same deposit (Chapter 2). This relationship is expressed by the equation

$$\text{Log } k = -2.97 \rho_d - 8.29 \quad (r^2=0.80),$$

para ρ_d entre 1.4 y 1.9 g/cm³

where *k* is the coefficient of permeability in m/s and ρ_d is the dry density in g/cm³.

For the average dry densities of the FEBEX tests, the coefficient of permeability calculated from the above equation, is 1.4 to 1.5 times the average coefficient determined from these tests; however,

differences of this magnitude are quite small in permeability testing. As can be seen in Table 4.19, the ratio between the coefficient of permeability for samples F-109 and F-2, for the dry density of 1.59 g/cm³, is 2.1 and these results came from the same set of tests made on the same dates.

UPC-DIT also made saturated hydraulic conductivity tests. First, the measurements were made directly in a specimen initially compacted to a dry density of 1.75 g/cm³ loaded in an oedometer at 5.0 MPa, and saturated. After saturation, the void ratio was 0.65 corresponding to a dry density of 1.64 g/cm³. Later, a pressure gradient of water was applied, with 0.50 MPa applied to the bottom of the sample and 0.05 MPa applied to the top. The pressures were maintained for 10 days to establish a steady state in the flux through the specimen. The pressures and volumes of water were measured with two GDS controllers. The total swelling of the specimen during the permeability test was less than 10 μm. The methodology utilized is described in Procedure 70-UPC-G-0-05 [71].

Figure 4.52 shows the inflows and outflows of water. Using the average flows in and out, the resulting permeability was 6.9 ·10⁻¹⁴ m/s.

The dry density of the specimen in this UPC-DIT test was 1.64 g/cm³ and the coefficient of permeability is 1.9 times greater than that obtained by

Table 4.19
Values of hydraulic conductivity of the tested specimens

Sample reference	Dry density g/cm ³	Initial w %	k 10 ⁻¹⁴ m/s	Dry density g/cm ³	Initial w %	k 10 ⁻¹⁴ m/s
F-2	1.59	14.8	3.1	1.70	14.8	3.8
F-18	1.60	15.0	5.9	1.70	15.1	2.9
F-37	1.60	13.3	6.0	1.70	13.4	3.8
F-75	1.61	11.7	5.9	1.69	11.7	3.2
F-109	1.59	12.5	6.4	1.70	11.9	4.1
F-119	1.58	14.0	8.3	1.67	14.5	3.7
AVERAGE	1.60	13.5	5.9	1.69	13.6	3.6
STD. DEV.	0.01	1.3	1.7	0.01	1.5	0.4

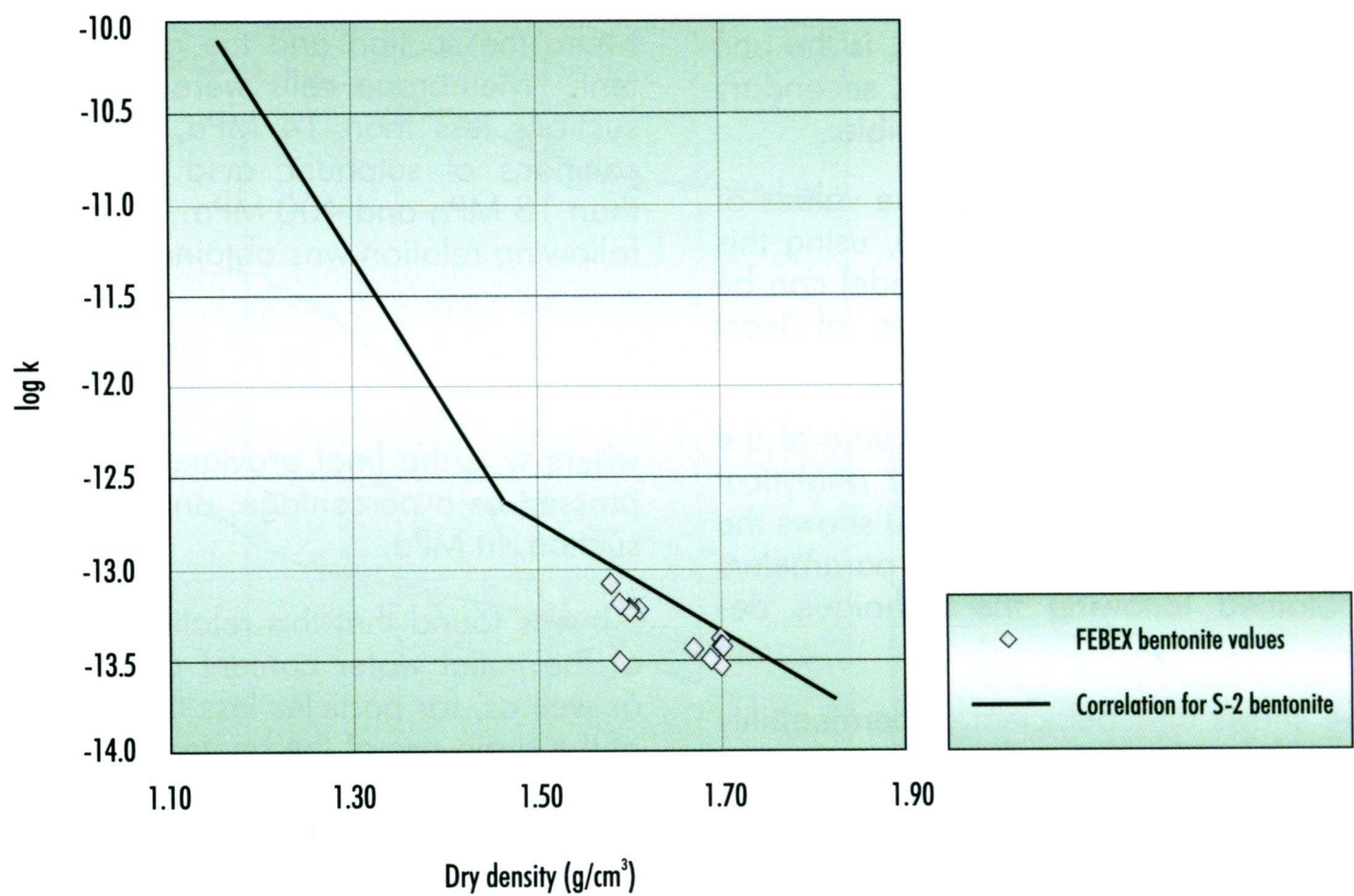


Figure 4.51: Hydraulic conductivity values of tested specimens and previous regression lines for bentonite S-2.

CIEMAT in the FEBEX set of tests for an average dry density of 1.69 g/cm³ and 1.2 times greater than that obtained for an average dry density of 1.60 g/cm³. Using the equation obtained by CIEMAT for S-2 bentonite, the coefficient of permeability calculated by UPC-DIT for a dry density of 1.64 g/cm³ is 6.9·10⁻¹⁴ m/s, which is the same than that determined in the test.

Although UPC-DIT made only one direct measurement test, two conclusions can be drawn from the result. The first conclusion is that there is good agreement between the results of both laboratories taking into account that scattering (even by a factor of 5) is usual in permeability testing of the same material in the same laboratory; thus, this agreement confers confidence in the results. The second conclusion is that the coefficients of permeability, of the specimens of the FEBEX bentonite tested by CIEMAT, show a tendency to be slightly less than the value measured in the prior tests made on samples from the same deposit, the variation being sufficiently small that it can be assumed that the most probable value for the average coefficient of permeability would be determined by integrating

all the results obtained to date. This seems to be confirmed by the UPC-DIT test.

UPC-DIT has also determined, after saturation, the coefficient of permeability by means of oedometer tests on the same specimens on which they had previously made the swelling under load tests. To calculate the saturated coefficient of permeability, the following analytical model was used to evaluate the relationship between strain and time.

$$\delta = \delta_0 + \frac{2H\Delta\sigma}{E_m} U(t, c_v, H) + 2HC_\alpha \log(t / t_{90});$$
$$c_v = \frac{kE_m}{\gamma_w}$$

where δ is the specimen settlement, δ_0 is the instantaneous compression due to bedding and device elastic compression, $2H$ is the height of the specimen, $\Delta\sigma$ is the stress increment, E_m is the confined modulus, $U(t, c_v, H)$ is the degree of consolidation derived from Terzaghi's theory, C_α is the secondary consolidation coefficient, t is time, t_{90} is

the time associated with 90% of primary consolidation, c_v is the coefficient of consolidation, k is the coefficient of water permeability, and γ_w is the unit weight of water. For times less than t_{90} , secondary consolidation was assumed to be negligible.

Minimizing the differences between the values of settlements –measured and calculated, using this model– a set of parameters for the model can be obtained by means of the technique of least squares.

Figure 4.53 shows the evolution with time of the measured vertical strains and the best analytical approximation for each test. Table 4.20 shows the data from the oedometer tests and the parameters that were obtained following the technique described.

Figure 4.54 shows the coefficient of permeability as a function of the dry density from the conventional oedometer tests and by direct measurement.

Suction/water content relationship

In the previous studies (Chapter 2), CIEMAT made a large number of tests, on samples of ben-

tonite extracted from the same deposit as the FEBEX bentonite, to determine the relationship between the suction and the gravimetric water content. Membrane cells were used for testing with suctions less than 14 MPa, and desiccators with solutions of sulphuric acid for suctions between 13 MPa and 400 MPa. From all the tests, the following relation was obtained

$$w = 36.1 - 12.0 \log s$$

where w is the final gravimetric water content, expressed as a percentage, and s is the value of the suction, in MPa.

It was found that this relationship is independent of the initial water content and initial dry density, as well as, for particles less than 5 mm and 2 mm, of the grain size of the bentonite.

To see if this relationship is true for the FEBEX bentonite, CIEMAT made another set of tests with samples F and A plus others from the homogenized sample 70-IMA-3-4-0. The tests were made in sulfuric acid desiccators, following Procedure 70-IMA-G-0-19 [72]. Nominal suctions of 260,

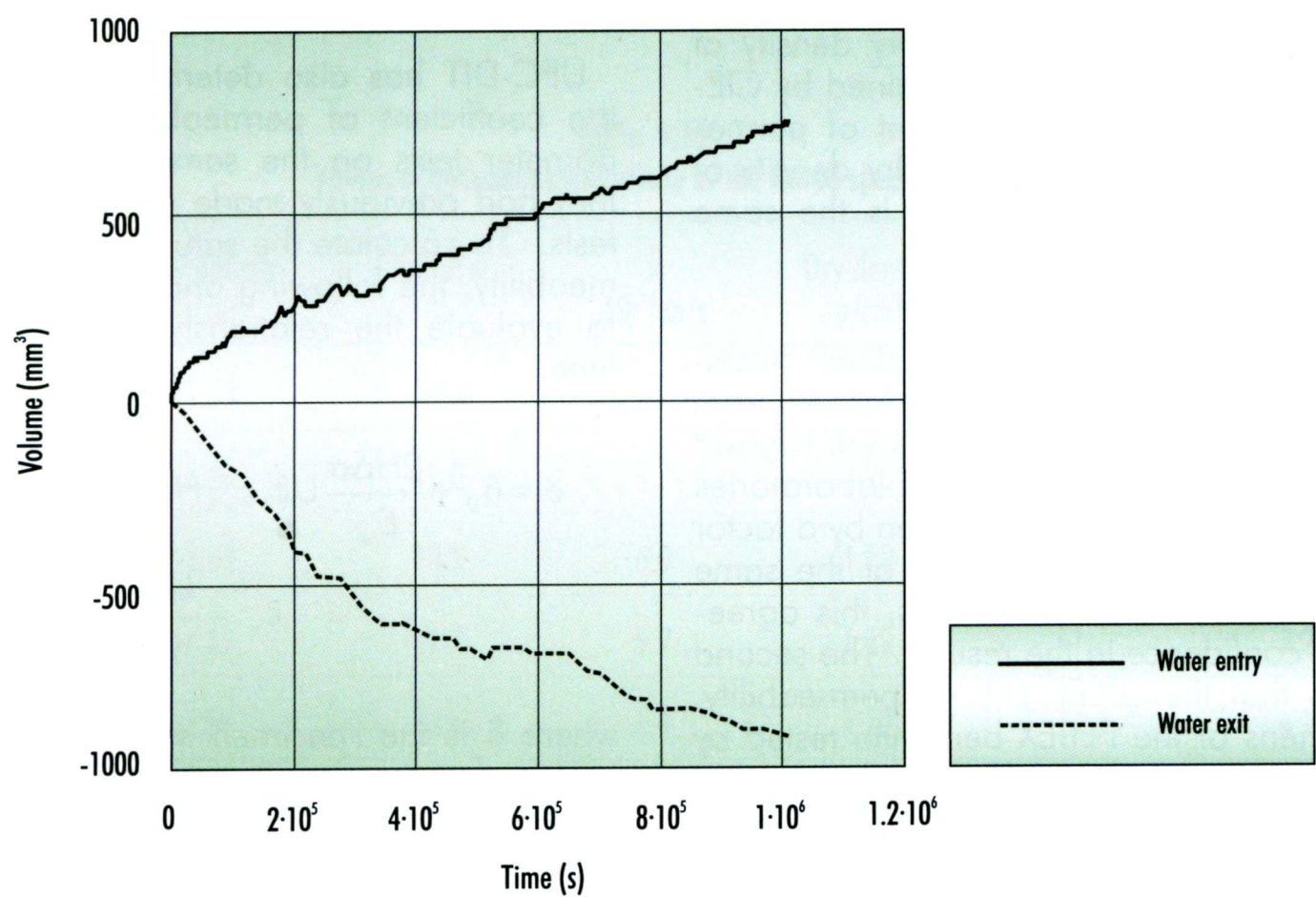


Figure 4.52: Evolution of water flow during permeability test.

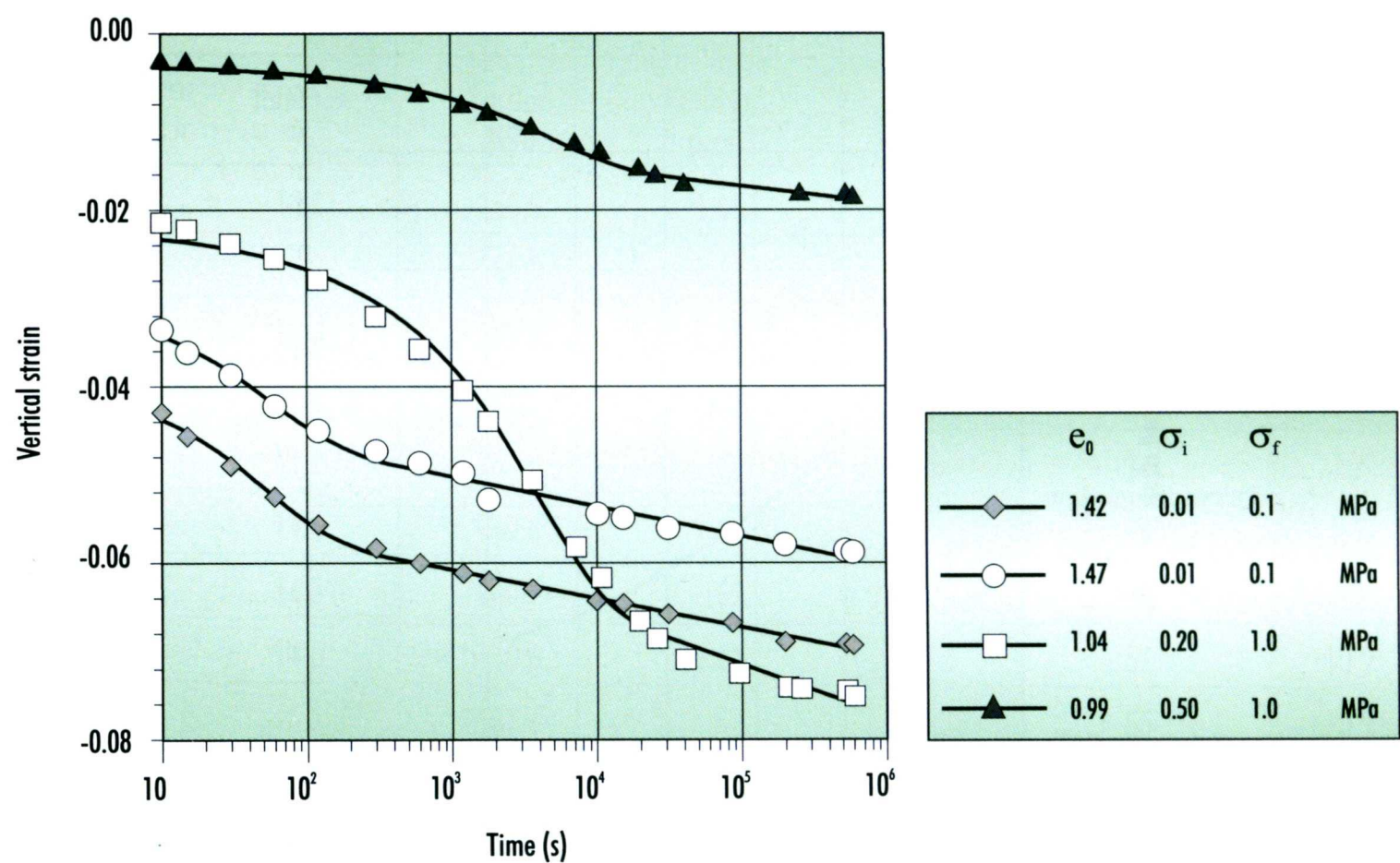


Figure 4.53: Evolution with time of vertical strains from oedometer tests.

140, and 14 MPa were applied on cylindrical specimens of 1.5 cm in diameter and 1.2 cm high, compacted to a nominal dry density of 1.60 g/cm³ with the water content existing in the storage bags (Table 4.1). The suction of 140 MPa corresponds, approximately, to the water content at equilibrium for the samples stored in the laboratory.

The initial and final water contents, the value of the suction, the final dry density, and the degree of saturation were determined for each specimen. The results obtained are presented in Table 4.21. Each determination was made in duplicate (two specimens were tested) and the results indicated in the table for each sample are the average value of

Table 4.20
Oedometer tests

ρ_d g/cm ³	e_i	σ_i MPa	σ_f MPa	c_v 10 ⁻³ cm ² /s	E_m MPa	k m/s	C_α 10 ⁻³	δ_0 μm	ε_γ μm
1.15	1.417	0.01	0.1	4.39	4.42	9.9·10 ⁻¹⁰	3.26	597	6,6
1.12	1.473	0.01	0.1	4.47	4.90	9.12·10 ⁻¹⁰	3.37	446	12.0
1.36	1.036	0.2	1.0	0.0415	18.27	2.27·10 ⁻¹²	6.00	273	15.4
1.39	0.999	0.5	1.0	0.0320	42.46	7.53·10 ⁻¹³	1.99	43	5.5

ρ_d = initial dry density; e_i = initial void ratio; σ_i = initial load; σ_f = final load; c_v = coefficient of consolidation; E_m = confined modulus; k = coefficient of permeability; C_α = secondary consolidation coefficient; δ_0 = instantaneous compression due to bedding and device elastic compression; ε_γ = average difference between the measured values and the analytical approximation.

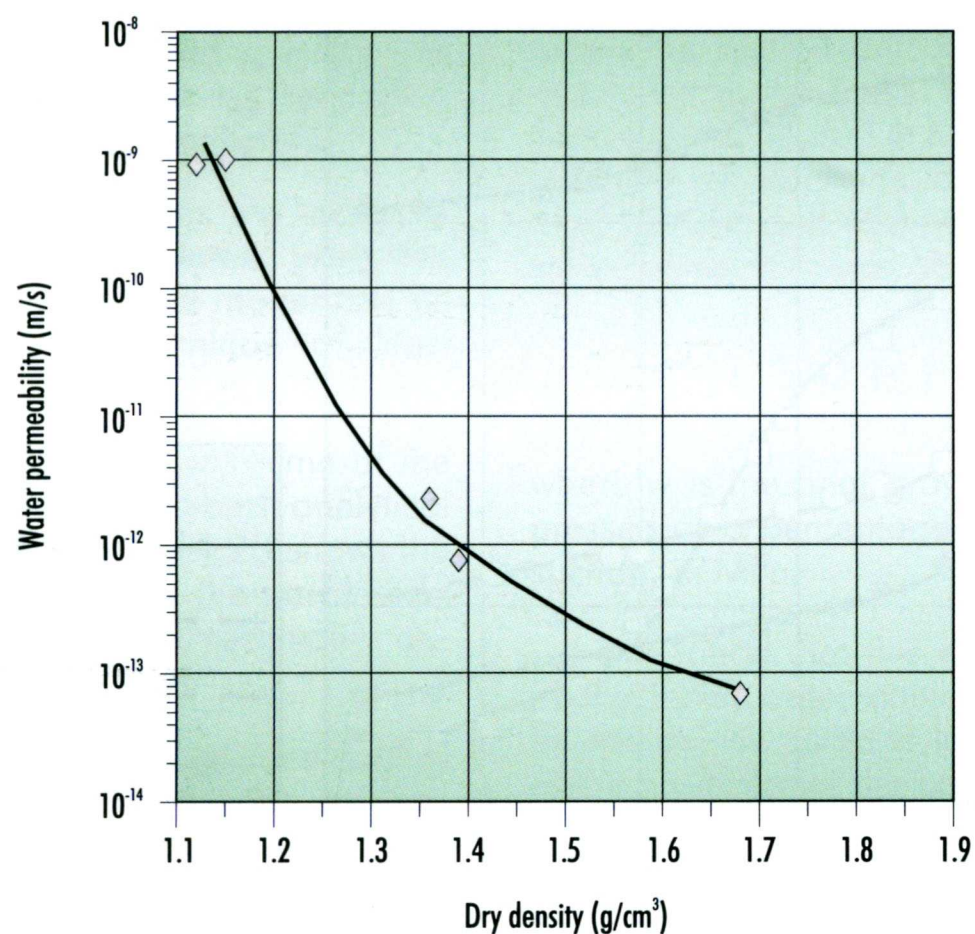


Figure 4.54: Coefficient of saturated water permeability, from the conventional oedometer tests, vs. dry density.

those two specimens. In the final column of the table are the values of the water contents calculated for each suction, utilizing the previous equation. Figure 4.55 shows the graphical representation of the previous equation and the results of the tests made on the FEBEX bentonite.

As can be observed, from the data in the table as well as in the figure, the water content of these tests practically coincides with the empirical relationship of the bentonite S-2 for the low suctions, whereas it was on the order of 10 % to 15% greater for the high suctions.

UPC-DIT also performed this type of test, following Procedure 70-UPC-G-0-03 [73]. Their tests were made with cylindrical specimens of diameter 1.5 cm and height of 1.2 cm, compacted to initial nominal dry densities of 1.53, 1.63, and 1.73 g/cm³.

The tests were made in hermetically sealed chambers at specified temperatures (27°C and 72°C and, in some cases, also at 40°C and 57°C) and subjected to a relative humidity of controlled air by means of sulphuric acid and sodium chloride at different concentrations. To avoid altering

the sample, the dry density corresponding to each suction was not determined.

Figure 4.56 shows the suction versus water content curves, with an indication of the initial dry density and temperature of the test. Figure 4.57 shows the set of all the results for the different densities and suctions. The effects of the initial density and of the temperature are very small; thus, it was not possible to quantify and separate these effects in the suction versus water content curve.

From all the data obtained, for water contents lower than 20 %, the suction versus water content relationship can be expressed by the equation

$$w = 40.7 - 14.3 \log s$$

where w is the final gravimetric water content, expressed in percent, and s is the suction, in MPa.

The experimental equations of both laboratories are very similar and, in fact, for the suction intervals of the tests, the water content differs by only 1 or 2 percentage points, and this is the same order of magnitude as the scattering that can be de-

Table 4.21
Suction / water content relationship

Sample reference	Initial w %	Suction MPa	Final ρ_d g/cm ³	Final w %	Final S _r %	w (*) %
F-2	14.8	255.5	1.65	9.0	39	7.8
F-17	16.0	255.5	1.61	9.0	36	7.8
F-37	13.3	255.5	1.63	9.1	38	7.8
F-59	12.7	255.5	1.63	9.2	38	7.8
F-75	11.7	260.6	1.62	8.3	34	7.7
A-1	14.4	260.6	1.65	8.4	36	7.7
A-5	14.5	260.6	1.65	8.3	35	7.7
F-2	14.8	138.6	1.61	13.2	52	10.9
F-17	16.0	138.6	1.62	13.3	55	10.9
F-37	13.3	138.6	1.60	13.0	51	10.9
F-59	12.7	138.6	1.60	12.8	49	10.9
F-75	11.7	144.1	1.60	10.9	43	10.8
F-109	11.9	143.3	1.62	11.1	45	10.8
F-119	12.3	143.3	1.63	11.5	47	10.8
A-1	14.4	144.1	1.63	11.7	48	10.8
A-5	14.5	144.1	1.62	11.6	48	10.8
70-IMA-3-4-0	13.1	143.3	1.62	11.5	47	10.8
F-109	11.9	126.6	1.60	12.1	48	11.4
F-119	12.3	126.6	1.59	12.7	49	11.4
70-IMA-3-4-0	13.1	126.6	1.60	12.3	48	11.4
F-2	14.8	14.5	1.39	22.7	65	22.5
F-17	16.0	14.5	1.43	22.6	69	22.5
F-37	13.3	14.5	1.35	22.6	61	22.5
F-59	12.7	14.5	1.36	22.8	62	22.5
F-75	11.7	14.0	1.35	21.9	59	22.6
F-109	11.9	14.9	1.35	21.9	59	22.3
F-119	12.3	14.9	1.35	22.2	60	22.3
A-1	14.4	14.0	1.34	22.4	60	22.6
A-5	14.5	14.0	1.35	22.4	61	22.6
70-IMA-3-4-0	13.1	14.9	1.36	22.0	60	22.3

(*) According to the relationship previously established for the bentonite S-2.

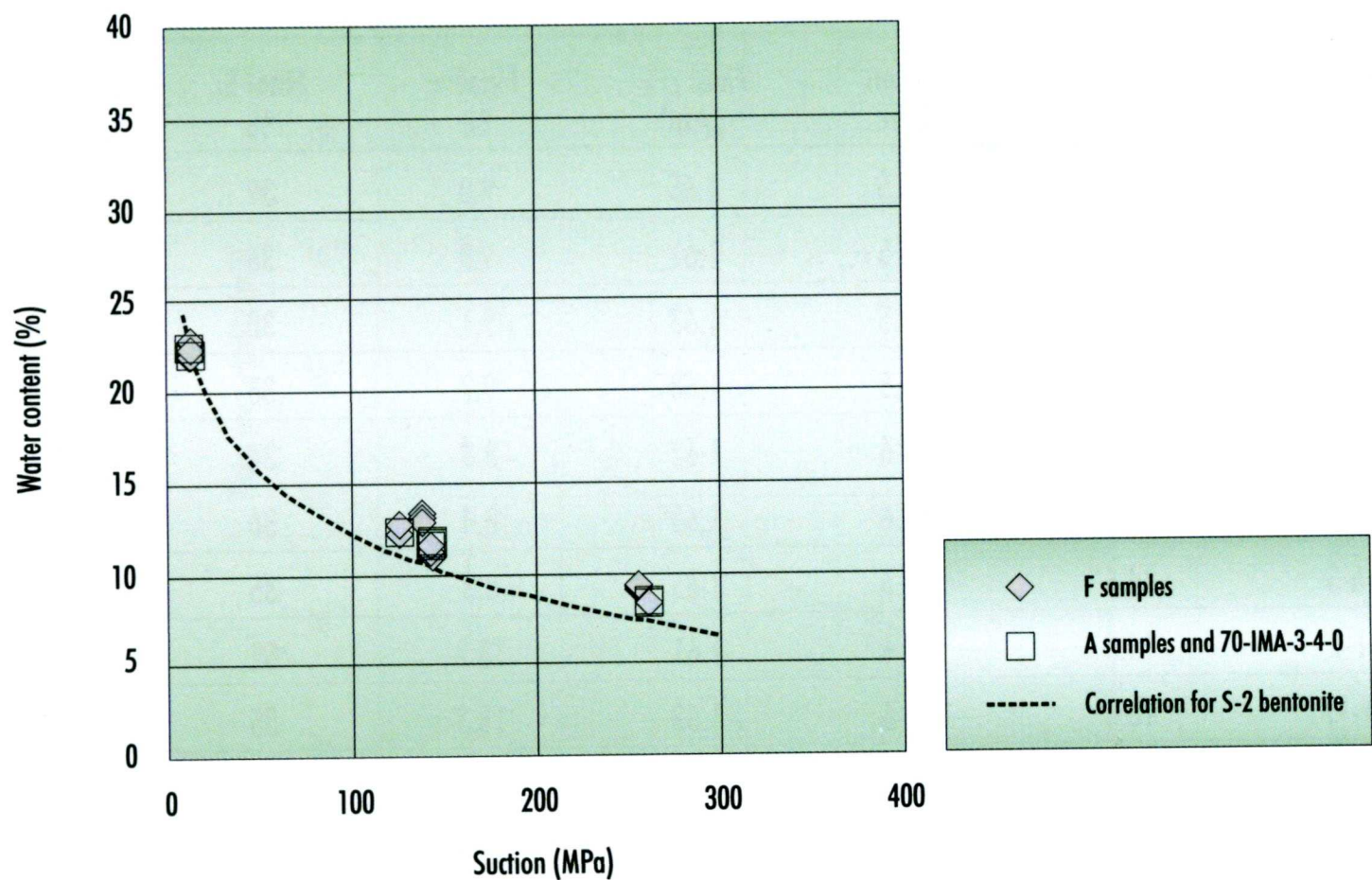


Figure 4.55: Relationship suction / water content.

tected in the same laboratory for different sets of tests on samples of the same bentonite.

4.4.3. Thermal tests

Thermal conductivity

The surficial thermal conductivity was measured with a probe placed on top of the compacted clay specimens, as is described in Procedure 70-IMA-G-0-13 [74].

The specimens were uniaxially compacted to nominal dry densities of 1.60 and 1.70 g/cm³ with water contents around 14 %, which correspond to a degree of saturation (S_r) of about 60 %, also with water contents about 26 % and 23 %, corresponding to a degree of saturation of 100 % for the dry densities of 1.60 and 1.70 g/cm³, respectively. Compaction pressures were on the order of 14 MPa, for dry densities of 1.60 g/cm³, and between 22 MPa and 30 MPa, for dry densities of 1.70 g/cm³. The conductivity was also measured in the same specimens after being dried at 100°C for 24

hours. The specimens were then weighed and measured to compare the change of density.

The tests were made on 37 specimens of 11 un-mixed samples (F samples) and in all cases the measurement was made by applying the probe to the compaction surface, so that the value obtained is the conductivity parallel to the direction of the compaction effort.

Table 4.22 presents the measured values of the thermal conductivity, the dry density, the water content, and the degree of saturation for each specimen tested.

Figure 4.58 shows, graphically, the values of the thermal conductivity (λ, W/m·K) in the direction parallel to the compaction effort as a function of the water content (w, %). A polynomial fit has been made, independent for each group of values, corresponding to the nominal dry densities of 1.60 g/cm³ and to those of 1.70 g/cm³. The equations of these lines are:

For a dry density of 1.60 g/cm³:

$$\lambda = 0.0013 w^2 - 0.0052 w + 0.5201 \quad (r^2=0.97; 18 \text{ points})$$

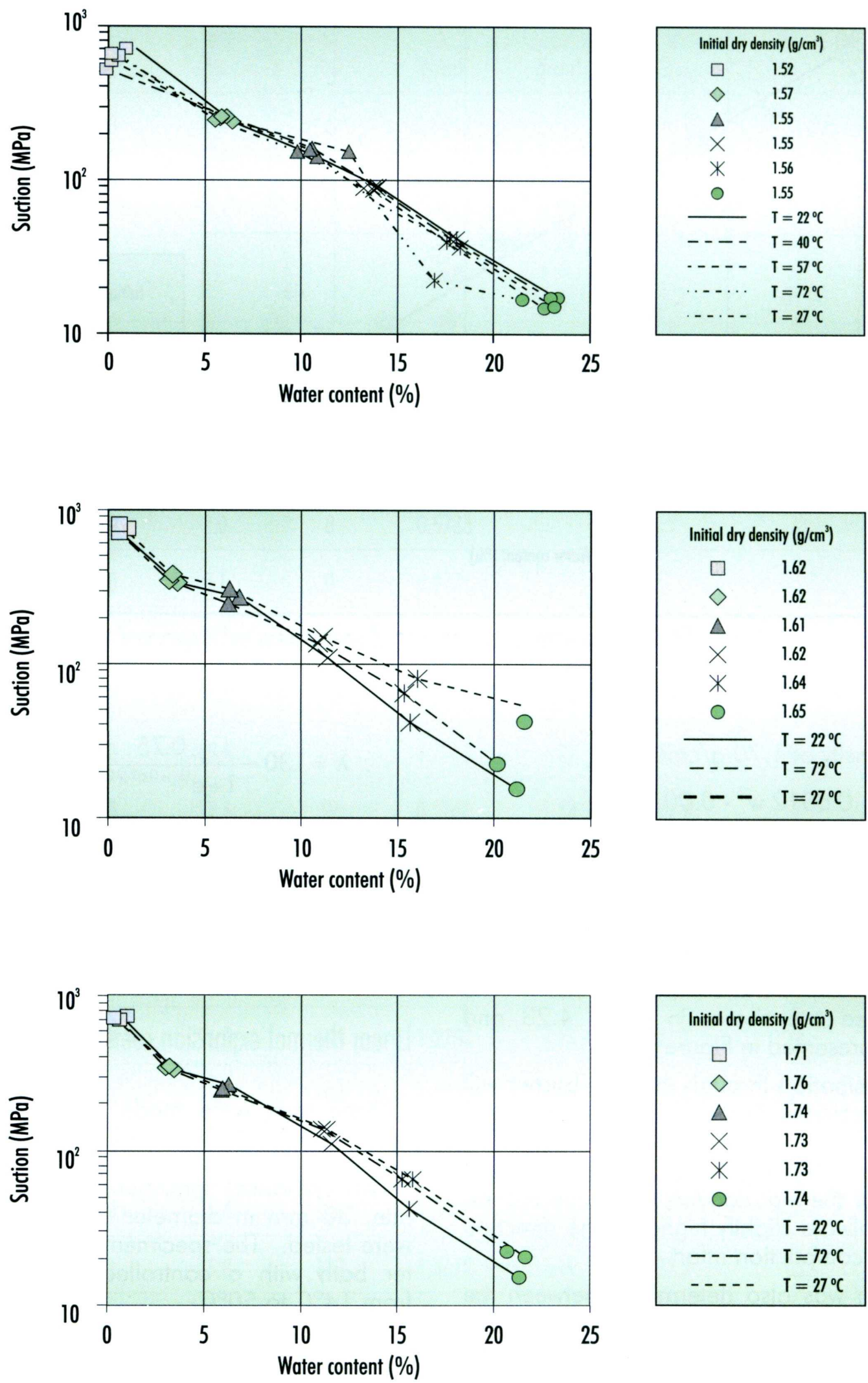


Figure 4.56: Suction / water content curves for different densities and temperatures.

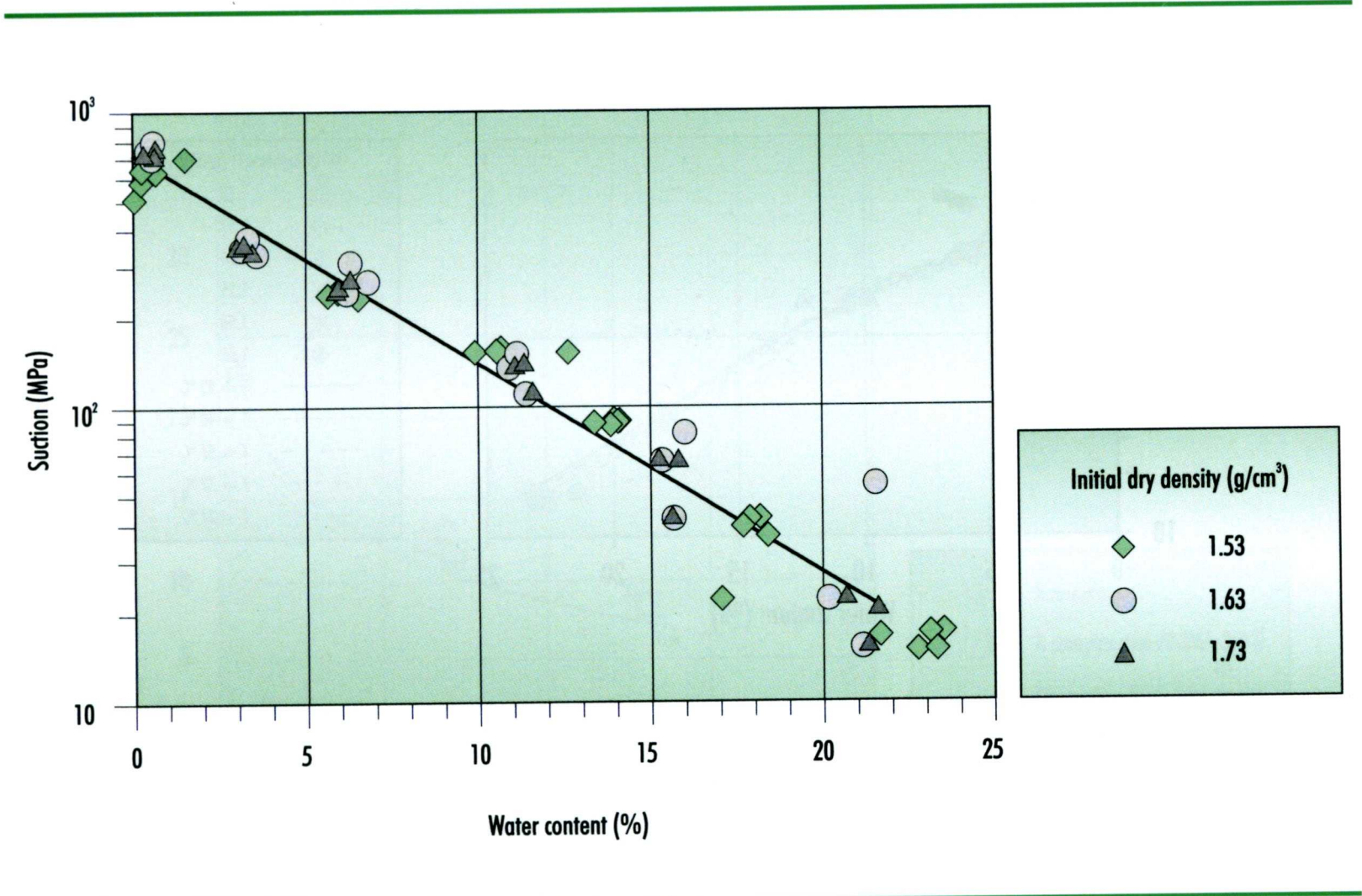


Figure 4.57: Suction vs. water content curves for different densities and temperatures.

For a dry density of 1.70 g/cm³:

$$\lambda = 0.0012 w^2 - 0.0016 w + 0.6237 \quad (r^2=0.97; 10 \text{ points})$$

In compacted specimens of the homogenized sample (70-IMA-3-4-0), the thermal conductivity was also measured but in both directions, parallel and perpendicular, to the compaction effort. The values obtained are shown in Table 4.23 and graphically represented in Figure 4.59.

The heat dissipation through the clay barrier will take place, principally, in the radial direction —perpendicular to the direction of the compaction effort on the blocks. Although there is no important variation in the thermal conductivity in the two directions, it is always slightly higher in the direction parallel to the compaction effort.

A correlation was also determined between the degree of saturation (S_r) and the measured values for the coefficient of thermal conductivity (λ) in the direction parallel to the compaction effort, for the unmixed samples (F samples) shown in Table 4.22 as well as for the homogenized sample 70-IMA-3A-0 shown in Table 4.23:

$$\lambda = 1.30 - \frac{0.75}{1 + e^{(S_r - 0.67)/0.11}} \quad 39 \text{ points}$$

In Figure 4.60 are represented the values of the thermal conductivity (λ , W/m·K), as a function of the degree of saturation (S_r) and the sigmoidal regression curve obtained, together with that obtained for the bentonite S-2.

Linear thermal expansion coefficient

These tests were made by UPC-DIT following Procedure 70-UPC-G-0-11 [75].

Two cylindrical specimens of compacted bentonite, 38 mm in diameter and 78 mm in height, were tested. The specimens were heated in a water bath with a controlled temperature ranging from 14°C to 50°C.

In Table 4.24 the characteristics of the specimens and the values of the resulting coefficient of linear thermal expansion are indicated. Figure 4.61 shows the measured strains, in the steady state, after each change of temperature.

Table 4.22
Thermal conductivity (λ) in direction parallel to compaction force

Sample reference	ρ_d g/cm ³	w %	S_r %	λ W/mK	ρ_d g/cm ³	w %	S_r %	λ W/mK
F-18	1.53	1.7	6	0.5013				
F-37	1.55	0.9	3	0.4797				
F-75					1.66	1.9	8	0.6195
F-75					1.67	0.1	0	0.6356
F-75					1.72	0.5	2	0.6293
F-109	1.53	0.1	0	0.4841				
F-18	1.60	1.0	4	0.5473				
F-37	1.60	2.0	8	0.5738				
F-109	1.58	0.1	0	0.4771				
F-119	1.59	1.1	4	0.5763				
F-2	1.59	15.5	60	0.6978	1.74	13.7	67	0.8992
F-18	1.60	15.5	61	0.7824	1.71	15.0	70	0.9402
F-37	1.63	12.6	52	0.7261	1.73	11.9	57	0.8339
F-75	1.60	13.1	51	0.6825	1.72	12.9	61	0.8077
F-102	1.59	15.4	60	0.6995				
F-119	1.60	13.3	52	0.6114	1.70	13.1	60	0.7923
F-2	1.62	26.0	105	1.2754	1.73	21.6	104	1.2183
F-17	1.63	25.3	104	1.2726	1.75	20.4	101	1.2450
F-37	1.62	26.2	106	1.3043	1.72	21.6	102	1.1247
F-59	1.64	25.4	106	1.3069	1.76	20.5	104	1.2342
F-75	1.61	25.8	103	1.3619	1.69	22.7	103	1.2717
F-79	1.61	27.1	108	1.2854	1.71	22.6	103	1.2541
F-102	1.59	27.4	106	1.2613	1.70	21.8	100	1.2709
F-119	1.60	26.6	104	1.2472	1.69	22.3	101	1.2531

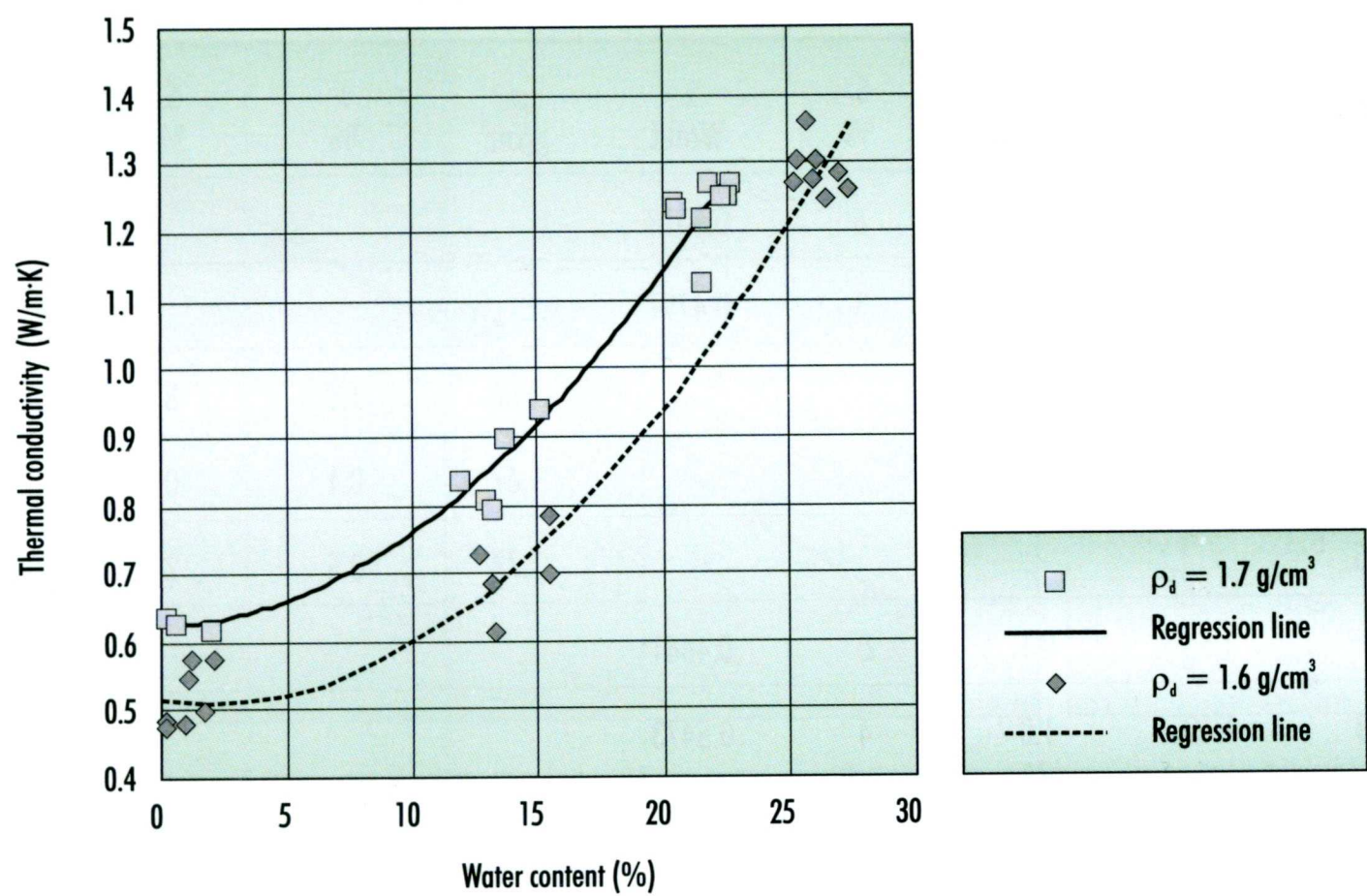


Figure 4.58: Thermal conductivity vs. water content, F samples.

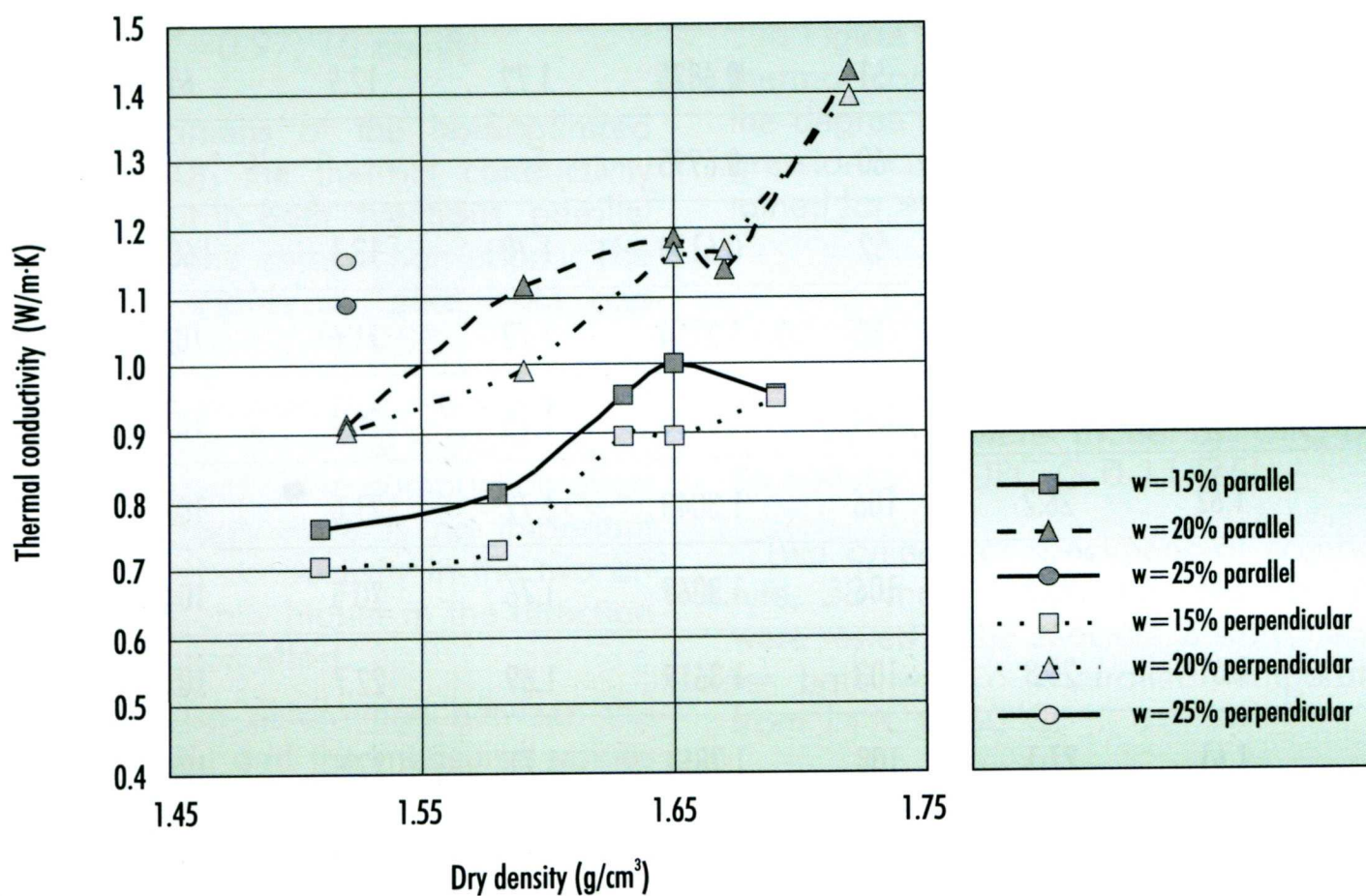


Figure 4.59: Thermal conductivity, measured in directions parallel and perpendicular to compaction effort, from sample 70-IMA-3-4-0.

Table 4.23
Thermal conductivity both parallel and perpendicular to compaction force in specimens compacted from sample 70-IMA-3-4-0

ρ_d g/cm ³	w %	S_r %	λ parallel W/mK	λ perpendicular W/mK
1.51	15	51	0.7621	0.7077
1.58	15	57	0.8114	0.7283
1.63	15	62	0.955	0.8934
1.65	15	64	1.0003	0.894
1.69	15	68	0.9531	0.9525
1.52	20	70	0.9128	0.903
1.59	20	77	1.1187	0.993
1.65	20	85	1.1851	1.1622
1.67	20	88	1.1405	1.1686
1.72	20	95	1.4319	1.3918
1.52	25	87	1.0913	1.1517

4.5. Sorption capacity

A set of laboratory batch sorption tests have been started, to check the behavior of some tracers under similar (although not identical) conditions to those expected in the "mock-up" and "in situ" tests. The tracers being used in these tests are chemical analogues of radionuclides which will be present in the radioactive waste, or stable forms of the same [76]. Depending on their sorption behavior, the two types of tracers are: conservative (those that will not be sorbed in the medium, thereby indicating water movement) and non-conservative (those that will move, suffering retention and/or retardation due to their sorption properties).

The tests were performed under oxic conditions using bentonite which had been ground to a grain size of less than 63 μm . Two different working temperatures (25°C and 90°C) were selected. The

time of contact was fixed at 7 days, the solid to liquid ratio was 1:10, and the initial concentration of each tracer was 200 $\mu\text{g/l}$. Constant stirring was maintained during each entire test. For the 25°C tests, a natural granite groundwater from El Berrocal Site was used, while for the 90°C tests, another granite groundwater of a similar chemical composition, but with a lower bromine content, was employed.

For the 25°C tests, polyethylene tubes were used to contain the samples; while to perform the 90°C tests, Pyrex glass tubes hermetically closed were used. When the boron was the tracer being tested, polyethylene tubes sealed with Teflon tape were used to avoid possible boron contamination from the glass.

The distribution relation (R_d , in ml/g) of an element is the ratio between the concentration of the element in the solid phase and the concentration in the liquid phase, in a test of a known duration in

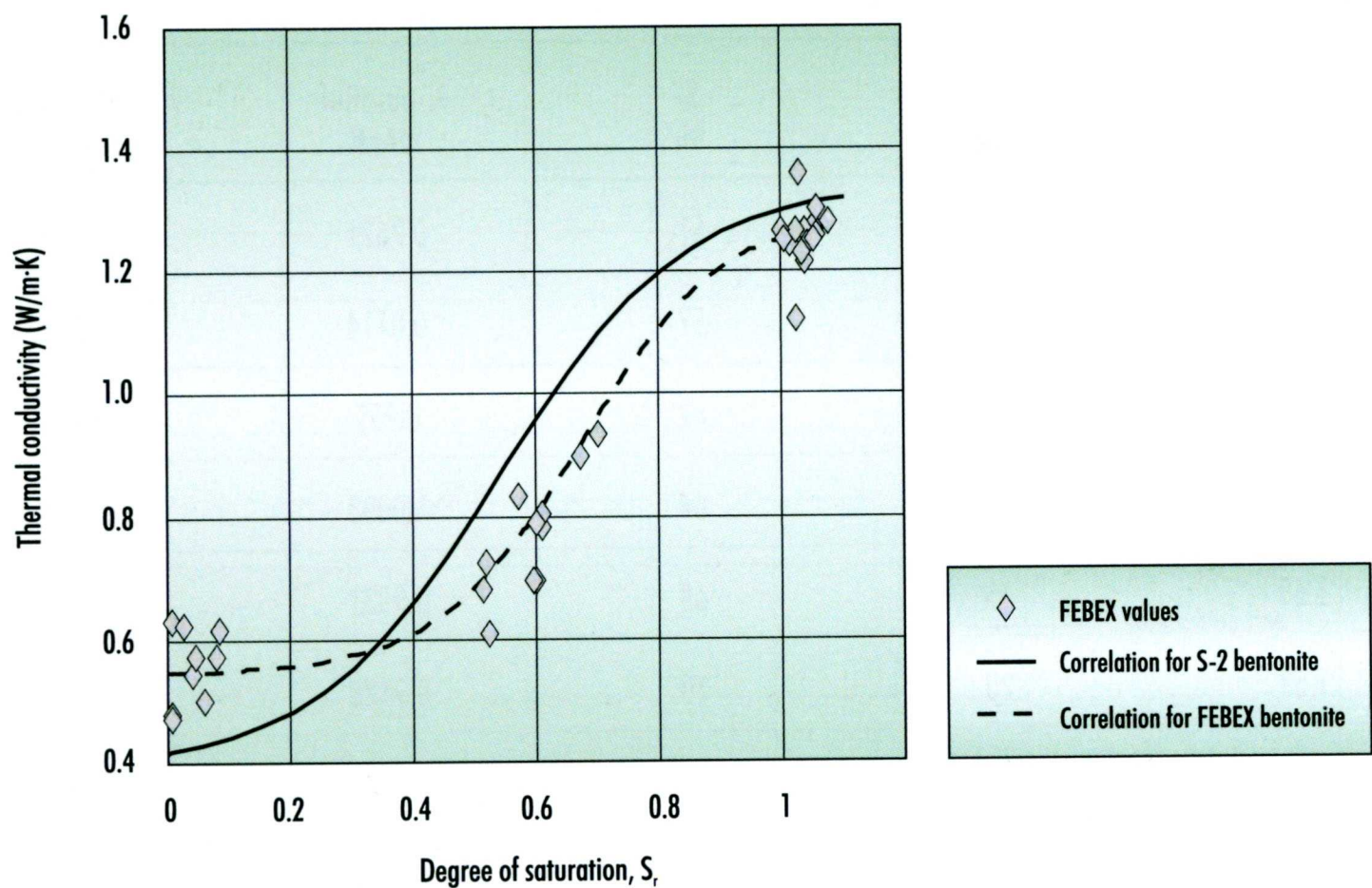


Figure 4.60: Thermal conductivity vs. degree of saturation from samples F and 70-IMA-3-4-0, and previous regression curve for bentonite S-2.

which the thermodynamic equilibrium has not been reached. The R_d of each tracer is given by:

$$R_d = \frac{C_o - C_f}{C_f} \cdot \frac{V}{M}$$

where C_o ($\mu\text{g/l}$) is the initial concentration of the tracer in the solution, C_f ($\mu\text{g/l}$) is the final concentration of the tracer in the solution, V (ml) is the volume of the employed solution, and M (g) is the clay mass. The R_d values obtained from the batch laboratory sorption tests are summarized in Table 4.25.

Iodine, selenate (VI), and perrhenate (VIII) behave as a non-sorbing (conservative) tracers, while the remaining elements behave as reactive (non-conservative) tracers

4.6. Microbiological characterization

The microbiological studies have commenced by detecting the presence of bacterial groups and es-

timating the number of bacteria whose activity could have influence on the properties of the clay (buffered capacity of pH, adsorption capacity, oxide-reduction state) or to produce, directly or indirectly, some corrosive substance (for example, SH_2). The studies made [30] have consisted in:

- To estimate the number of the following groups of bacteria: aerobic heterotrophic, anaerobic (forced and facultative) heterotrophic, microaerophilic, sulfate reducing, autotrophic, sulfure oxidizing, autotrophic sulfur oxidizing, ammonium oxidizing, nitrite oxidizing, iron oxidizing (*Leptothrix*, *Thiobacillus ferrooxidans*).
- To count and make a morphological study of the distinct bacterial colonies in specific solid media.
- To estimate the metabolic bacterial activity, with $[^{14}\text{C-U}]$ -glucose, in all the groups of heterotrophic bacteria.
- To determine, by analysis, the presence of iron reducing bacteria in the clay.



Table 4.24
Table 4.24: Thermal expansion tests

Specimen	Initial dry density g/cm ³	Water content %	Temperature path °C	Linear thermal expansion coefficient 1/°C
090996a	1.68	14.42	22.6-14.8	$5 \cdot 10^{-5}$
090996a	1.68	14.42	16.6-41	$8 \cdot 10^{-5}$
120996e	1.71	12.53	31-51	$21 \cdot 10^{-5}$
120996e	1.71	12.53	51-41	$6 \cdot 10^{-5}$

The extraction of the sample in the quarry, as well as later conditioning (homogenizing and drying), were not undertaken under sterile conditions; thus, the microorganisms present in the sample are a mixture of the natural ones and those introduced in the conditioning of the clay.

The samples were stored and handled in sterile conditions so as not to introduce other microorganisms, beyond those in the clay "in situ" and

those introduced during extraction and conditioning (drying, transporting, etc).

To determine the presence of the bacterial types, the clay samples are put in direct contact with different culture media. The culture media being employed as well as the procedure being followed are described in detail in [30]. The estimation of the number of bacteria of the different groups is being made by the method of most probable number.

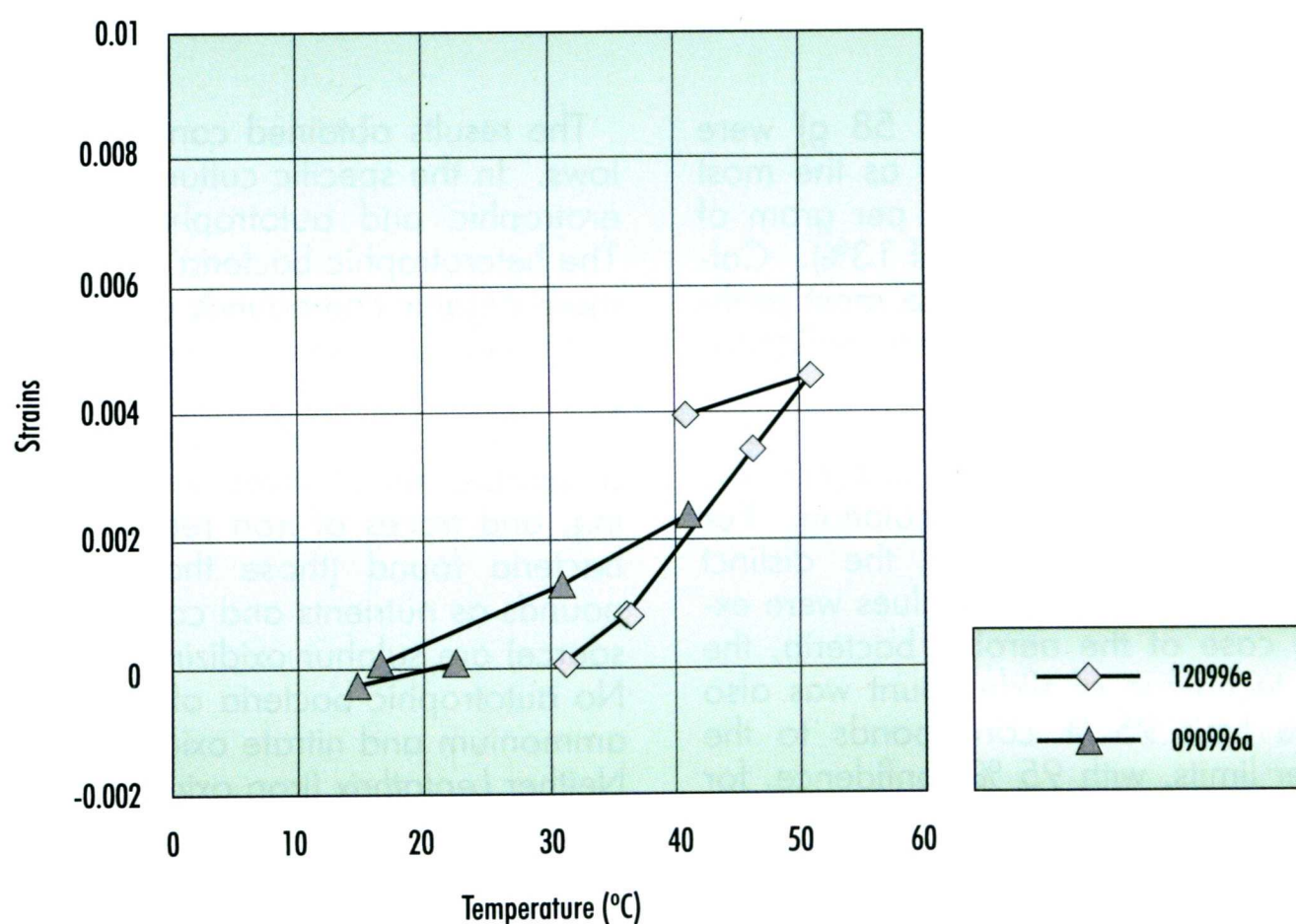


Figure 4.61: Results of thermal expansion tests.

Table 4.25
Batch R_d and sorption results

Element	25°C		90°C	
	R _d ml/g	Sorption %	R _d ml/g	Sorption %
I	1.9	16.5	0.2	2
Re	0	0	0	0
Se	0 (a)	0 (a)	0	0
B	22.5	69.3	38.7	79.5
Cs	≥ 1 990	≥ 99	≥ 1 990	≥ 99
Sr	90	90	98	91
La	≥ 1 990	≥ 99	≥ 1 990	≥ 99
Nd	≥ 1 990	≥ 99	≥ 1 990	≥ 99
Eu	≥ 1 990	≥ 99	≥ 1 990	≥ 99
Th	≥ 1 990	≥ 99	≥ 1 990	≥ 99
U	1.1	9.8	≥ 920	≥ 98

(a) Experiments performed by using the same granitic groundwater as in the 90°C batch experiments.

Seven samples (between 5 g and 58 g) were analyzed. The results are expressed as the most probable number (MPN) of bacteria per gram of clay analyzed (with a water content of 13%). Calculating the arithmetic average of the most probable numbers and of the corresponding confidence limits in the different samples analyzed, the values that were obtained are shown in Table 4.26. The number between the parentheses indicate the number of samples used for the calculation. For the calculation of the average of the distinct groups, the highest and the lowest values were excluded. In the case of the aerobic bacteria, the number by the technique of slide count was also estimated. The limit 95 % corresponds to the lower and upper limits, with 95 % confidence, for the estimated most probable number. The sign (-) indicates that no bacteria were detected.

The results obtained can be summarized as follows. In the specific culture media are found heterotrophic and autotrophic bacteria and fungi. The heterotrophic bacteria (those that need one or more organic compounds as carbon source) found in greater numbers are aerobics. The *Streptomyces* genus has been identified. Other heterotrophic bacteria groups detected were: microaerophilic, anaerobic (facultatives and strict), sulfate reducing, and traces of iron reducing. The autotrophic bacteria found (those that use inorganic compounds as nutrients and carbon dioxide as carbon source) are sulphur oxidizing and sulfide oxidizing. No autotrophic bacteria of the nitrogen cycle, nor ammonium and nitrate oxidizing have been found. Neither *Leptothrix* (iron oxidizing at neutral pH) nor *Thiobacillus ferrooxidans* (iron oxidizing at acidic and neutral pH) have been found.

Table 4.26
Counting of the different groups of bacteria found in 70-IMA-3-4-0 bentonite

Type of bacteria	MPN
Aerobic Limit 95 %	100 000 (6) 18 000 - 363 000
Aerobic (slide)	90 000 (5)
Anaerobic Limit 95 %	1 400 (4) 260 - 5 600
Microaerophilic Limit 95 %	78 200 (5) 12 000 - 398 000
Sulfur oxidizing Limit 95 %	2 600 (5) 400- 11 000
Sulfide oxidizing Limit 95 %	28 (4) 2 - 140
Sulfate reducing Limit 95 %	48 (4) 8 - 200
Nitrobacter	- (6)
Nitrosomonas	- (6)
<i>Tiobacillus ferroxidans</i>	- (2)

5

DESIGN, FABRICATION, AND CHARACTERISTICS OF THE BENTONITE BLOCKS

5.1. General

The blocks for constructing the clay barriers were fabricated in Spain: for the “mock-up” test by Jesus Castro, S. L. (Torrejon de Ardoz, Madrid), under the direction and control of CIEMAT; and for the “in situ” test by Refracta (Quart de Poblet, Valencia), under the direction and control of AITEMIN.

5.2. “Mock-up” test

In the “mock-up” test, the drift of a repository for HLW is being simulated in the laboratory of CIEMAT (Madrid) by a confining structure with interior dimensions: length of 6.00 m and diameter of 1.62 m. Two heaters, to simulate both the thermal effects of the waste decay and the waste containers, are centered in the structure with a distance between them of 0.75 m. Each heater has a length of 1.625 m and an exterior diameter of 0.340 m. Surrounding the heaters and filling the confining structure is a clay barrier formed by highly-compacted bentonite blocks. Figures 5.1, and 5.2 show, schematically, a longitudinal and two cross sections of the arrangement of the blocks within the confining structure.

The fabrication of the bentonite blocks for the clay barrier required the following steps: design of the blocks, fabrication of molds for producing the blocks, fabrication of the blocks themselves, and packaging and storing the blocks. All these steps are described in detail in the Technical Report 70-IMA-L-3-29 *Fabricación de bloques para el ensayo en maqueta del Proyecto FEBEX* [77].

5.2.1. Design of the blocks

To create a clay barrier of highly-compacted bentonite blocks, it was necessary to design the blocks, defining the geometry and the physical properties (dry density and water content), as well as to determine the quantity of blocks necessary to construct the barrier.

The geometry of the blocks was defined taking into account the principle to minimize the volume of gaps in the final clay barrier, to reduce the number of blocks to fabricate, and to avoid compaction surfaces greater than 0.1 m² (because of the limitation of the press force). Thus, it was decided to have the arrangement in two concentric crowns, with an interior core in the non-heater

zones (Figure 5.2). This arrangement was made with four types of blocks: A, B, C, and E, which are schematically represented in Figures 5.3.

A and B blocks were fabricated by compacting the bentonite directly into specially designed molds, whereas C blocks resulted from mechanically altering B blocks to form a flat inner surface (Figure 5.3). After the fabrication of all of the B blocks, the mold was modified, to eliminate the ears, in order to form an intermediate block from which the E blocks, of semi-hexagonal form, resulted.

The outer crown was formed with 12 A blocks and the inner crown with 6 blocks, of type B for the heater zones and of type C for the non-heater zones, with the core in the non-heater zones formed by 2 E blocks. (Figures 5.2).

After defining the dimensions of the blocks in the barrier cross section, the weight was set at about 25 kg, to facilitate the manual handling of the blocks. Table 5.1 shows the nominal design dimensions for each block type.

Considering that the interior length of the confining structure is 6.00 m and that the thickness of each block is 0.125 m, then the clay barrier would require 48 slices, 26 of which would be in the zones of the heaters. A total of 908 blocks would be required divided among the following types: 576 of A, 156 of B, 132 of C, and 44 of E. To compensate for possible damage during fabrication, transportation, or installation, 1000 blocks were fabricated, or about 10 % more than theoretically needed.

The nominal average dry density of the clay barrier should be, as a maximum, equal to the reference concept (1.65 g/cm³) or corresponding to a swelling pressure on the order of 6.0 MPa according to the characterization tests made prior to FEBEX. The clay barrier initially would have gaps between the blocks and the heaters, the confining structure, and the other blocks. It was estimated that the total volume of gaps would be on the order of 6% to 10% of the total volume to be filled and thus it was decided to fabricate the blocks to a nominal dry density of 1.75 g/cm³ to compensate.

In the studies prior to FEBEX (Chapter 2), it was determined that the optimum water content for compaction of this bentonite would be about 14 %, and that uniaxial compaction would provide the best results for the pressure to density relationship.

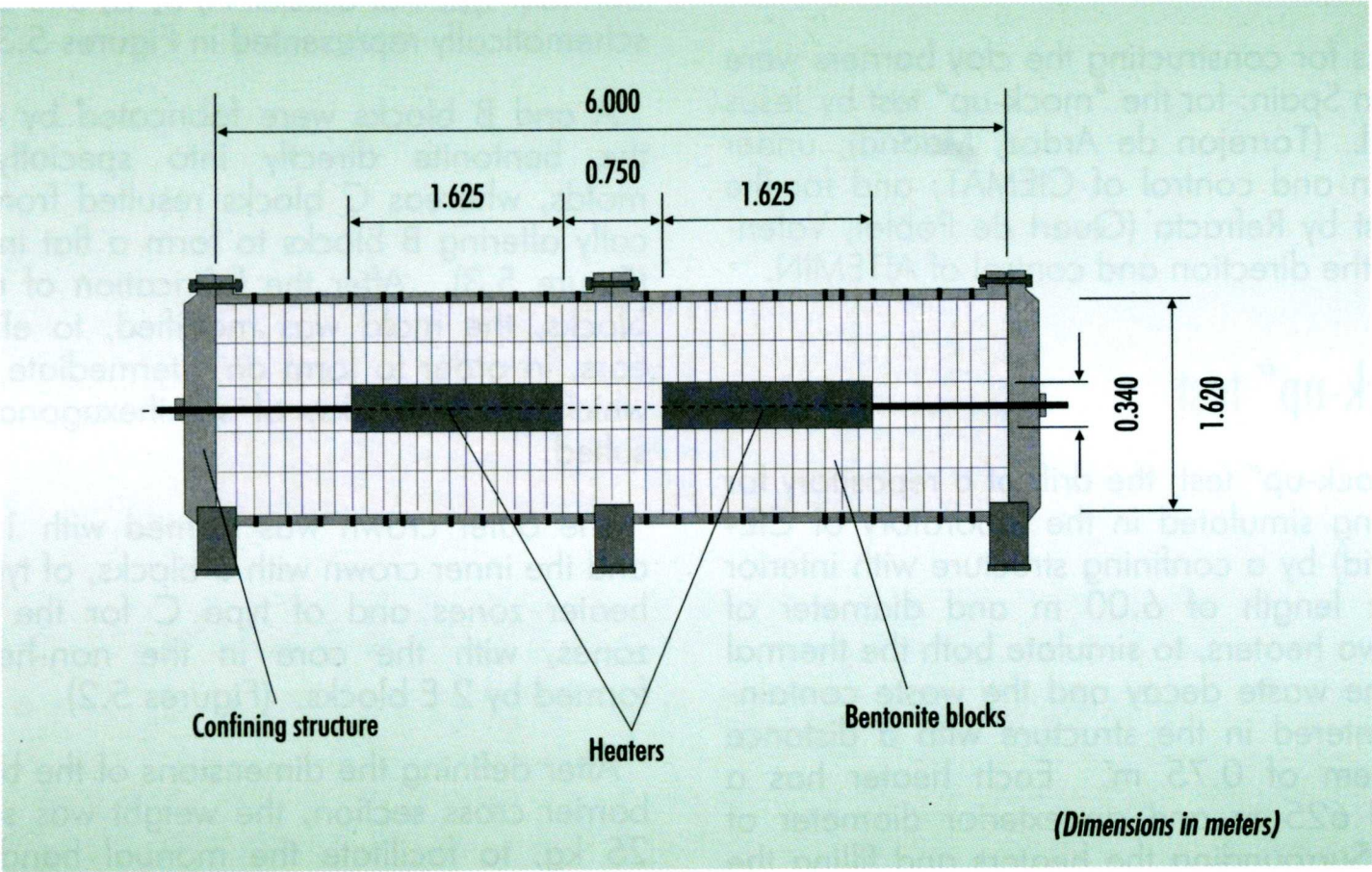


Figure 5.1: “Mock-up”: longitudinal section.

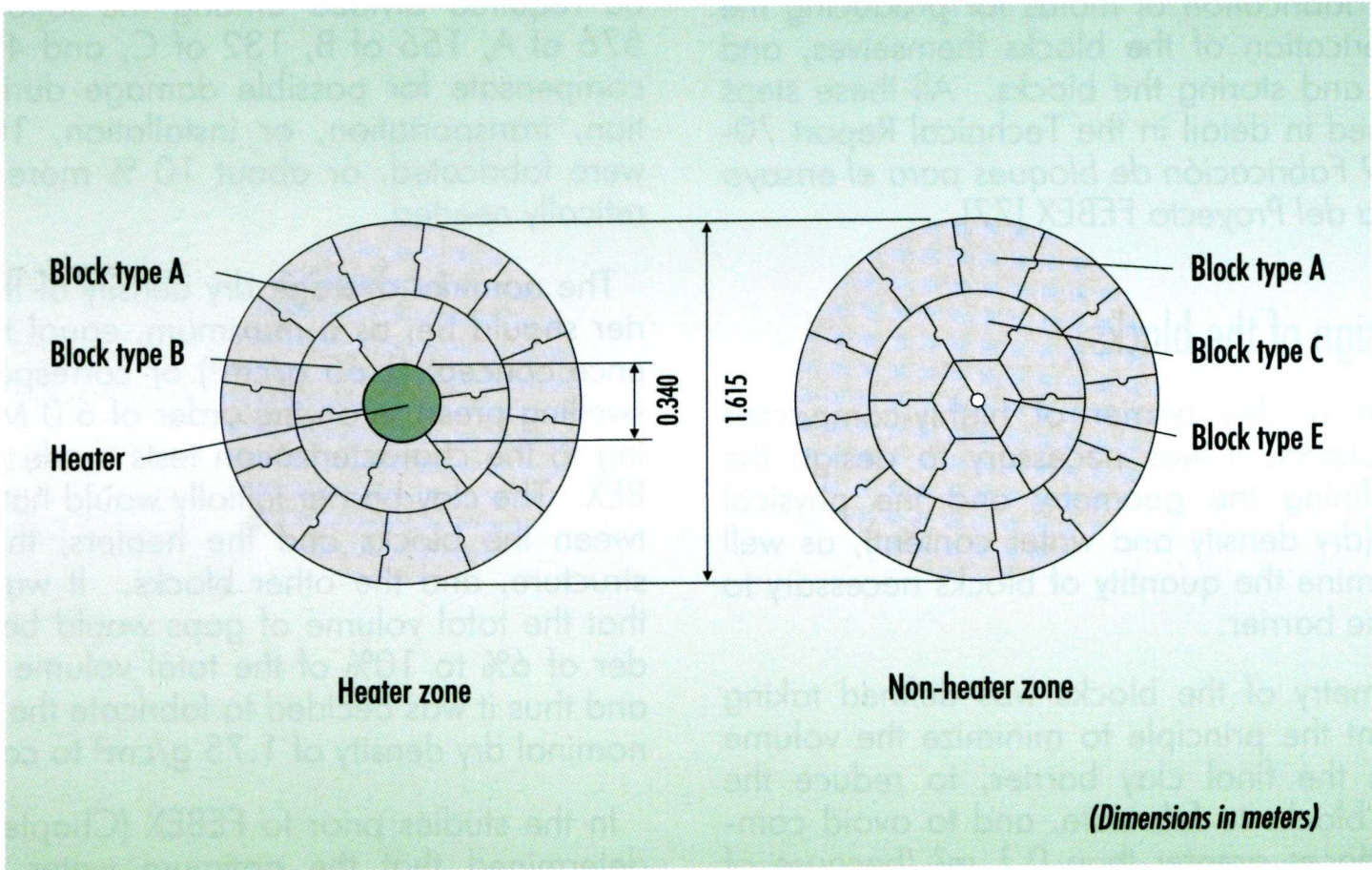


Figure 5.2: “Mock-up”: cross-section.

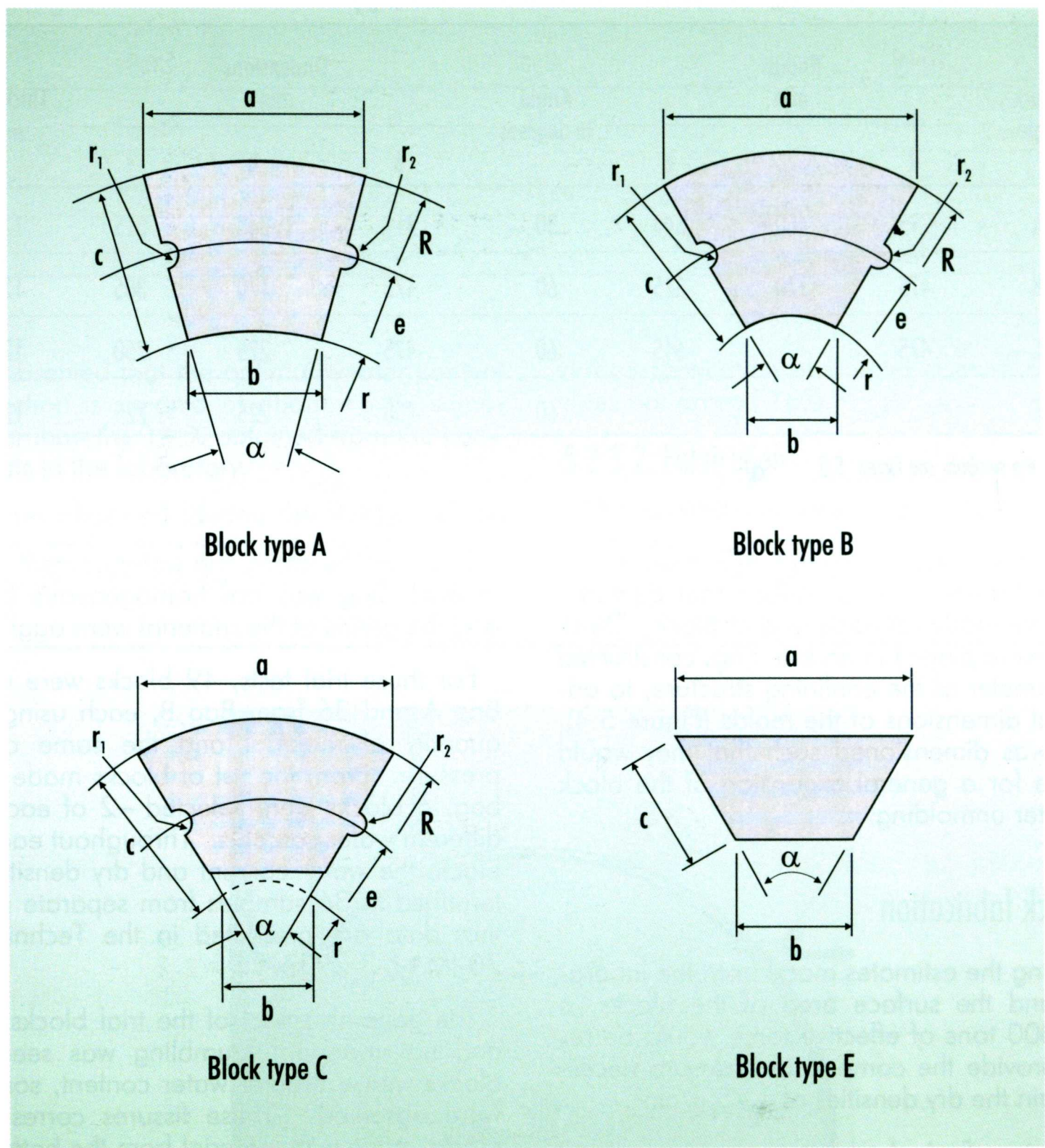


Figure 5.3: Schematic representation of blocks A, B, C y E.

In the laboratory, pressures between 45 MPa and 60 MPa were required to obtain, by uniaxial compaction, a dry density of 1.75 g/cm³.

5.2.2. Mold fabrication

The molds were designed and fabricated of carbon steel, taking into consideration the working conditions, the pressures required, etc. Jesus Cas-

tro, S. L. (a company fundamentally dedicated to die casting) made the molds as well as the blocks.

As was indicated in the previous section, it was only necessary to make two types of molds (for blocks A and B). Then, after all the B blocks were completed, the mold was modified for the fabrication of the intermediate blocks (called D) from which the final E blocks were mechanically fashioned.

Table 5.1
Nominal dimensions of the blocks (*)

Block type	Radius mm			Angle in degrees	Dimensions mm			Thickness mm
	R	r	e		a	b	c	
A	795	480	645	30	411	248	315	125
B	475	170	325	60	475	170	305	125
C	475		645	60	475	225	250	125
E				60	450	225	225	125

(*) For the symbols, see Figure 5.3.

After the design was completed, prototypes of the molds were fabricated and various trial compaction runs were made for each type of block. These trial pieces were placed in an iron ring, constructed with the diameter of the confining structure, to adjust the final dimensions of the molds (Figure 5.4). The mold was dimensioned such that they would compensate for a general expansion of the block of 1.5 % after unmolding.

5.2.3. Block fabrication

Considering the estimates made from the laboratory tests and the surface area of the blocks, a press of 1000 tons of effective force would be required to provide the compaction pressure necessary to obtain the dry densities of 1.75 g/cm³.

Jesus Castro, S. L. had equipment with these characteristics: a single-acting hydraulic press, able to supply an effective force of 1000 tons, with a lower piston (having an internal diameter of 780 mm) that acts against the upper frame, and a maximum working pressure in the hydraulic system of 20 MPa.

5.2.3.1. Trial tests prior to FEBEX

With the prototypes of the molds mentioned in the previous section, a set of compaction trial tests were made. For these tests, material was used which was from the same deposit as the FEBEX bentonite, but which had been stored, in the open air, in two big-bags (A and B) during a period of

four years; therefore, the water content distribution in each bag was not homogeneous (Table 5.2) and the grains of the material were agglomerated.

For these trial tests, 49 blocks were made from Bag A and 36 from Bag B, each using the same quantity of material and the same compaction pressure. From the set of blocks made from each bag, 6 blocks were selected –2 of each of the 3 different water contents. Throughout each selected block, the water content and dry density were determined in 36 samples from separate places and that data are presented in the Technical Report 70-IMA-L-3-29 [77].

The general aspect of the trial blocks was good and no important crumbling was seen. In the blocks with a greater water content, some fissures were observed. These fissures corresponded to blocks made with material from the bottom of both bags, as well as some from the middle part of Bag B, and is the result of shrinkage from the loss of moisture by the bentonite blocks during their storage.

In these trial blocks, no great variations were observed in the interior of the blocks and the average dry density obtained was 1.62 ± 0.01 g/cm³. There were somewhat higher dry densities obtained in the top part of the blocks and especially in the faces –interior and exterior– which could be attributed to the friction produced between the piston and the mold during the compaction process.

Figure 5.5 shows the average value of the dry density, for the 36 samples obtained in each trial block, as a function of the average water content.

Table 5.2
Water contents (%) of the bentonite used in the compaction trial test

Parts	Bag A	Bag B
Top part	11	15
Middle part	12	19
Bottom part	26	38

It can be observed that the optimum water content for compaction is situated at about 16 %, somewhat higher than the 14 % obtained from the compaction tests in the laboratory.

The fissures observed (during the storage) in the blocks fabricated with higher water contents indicate that caution should be taken to ensure that,

during fabrication, the water content of the blocks does not exceed 16%.

5.2.3.2. Fabrication

The final blocks were fabricated with the FEBEX bentonite (Chapter 3 and 4), with the shape and nominal dimensions indicated in Figure 5.3 and

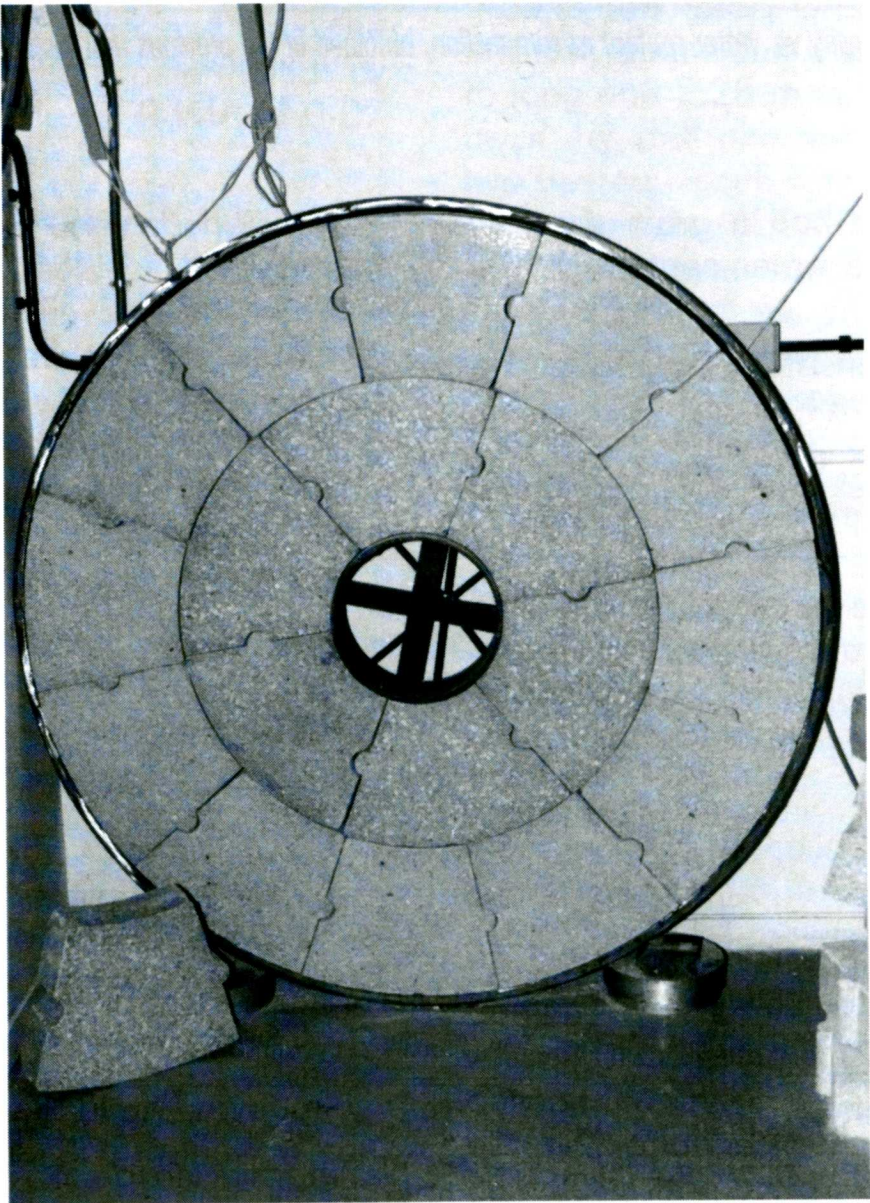


Figure 5.4: Adjustment test between blocks to define mold dimensions.

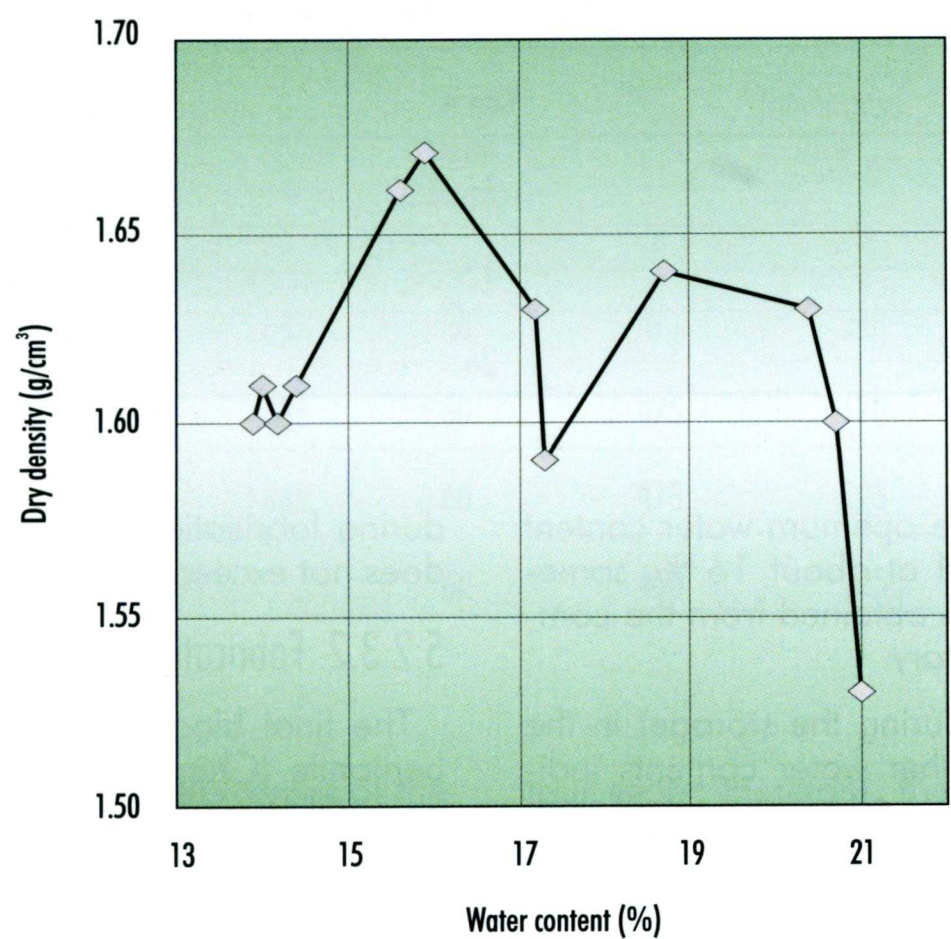


Figure 5.5: Average density vs. water content at compaction, obtained in the previous trial tests for compaction of blocks.

Table 5.1. The bentonite had a grain size less than 5 mm and an average water content on the order of 14 %.

The fabrication process consisted of the following activities:

1. Weighing the clay mass, which was calculated as a function of the water content and the type of block.
2. Placing the clay in the compaction mold by means of dropping from a hopper.
3. Spreading the material around in the mold to prevent weak zones in the edges of the blocks.
4. Compacting the bentonite by the specified pressure (40 to 50 MPa) using a pressure switch on the hydraulic system (15 to 18 MPa) for three successive cycles.
5. Mechanically unmolding and manually removing the block.
6. Visually observing and controlling the dimensions of the block –accepted or rejected.

The quality control of the fabrication in the factory followed the stipulations in Procedure 70-IMA-G-0-75 [78] and was made daily. Each block

was weighed and measurements made of the thickness on 4 sides as well as of the dimension between the side curves, (R - r), for the top and bottom faces. The weights, dimensions, evaluation of the visual aspect, dry densities, and water contents of all the blocks can be found in the Technical Report 70-IMA-L-3-29 [77].

By compaction, 598 A blocks were fabricated, 322 B blocks (from which C blocks were obtained), and 92 of the intermediate type of which all, by mechanical means, became E blocks. Table 5.3 shows a summary of the characteristics of the types of blocks used in the construction of the clay barrier

5.2.4. Block packaging and storing

The accepted blocks, in groups of 24, were placed in wooden boxes which had been lined with a plastic sheet. The boxes were loaded on pallets for transportation (Procedure 70-IMA-G-0-77 [79]).

Each box was considered a fabrication lot and, following Procedure 70-IMA-G-0-75 [78], the lot was assigned an independent control sheet show-

ing the characteristics (mass, dimensions, and aspect) of the blocks.

After fabrication and packaging, the blocks were transported to CIEMAT where they were stored until they were placed into the confining structure of the “mock-up” test.

5.2.5. Block characterization

Following the specifications in Procedure 70-IMA-G-0-76 [80] for the characterization of the bentonite blocks, a systematic sampling has been made by taking one of each 100 blocks fabricated, to determine and analyze the homogeneity of their characteristics. In each one of the blocks selected has been determined the thermal conductivity in the directions both parallel and perpendicular to the compaction force. Afterwards, the block was cut to obtain 15 samples, distributed among its upper, middle, and lower parts. On each one of these samples, the dry density and the water content, by the method of displacement of Hg (Procedure 70-IMA-G-0-10 [81]), have been determined. In addition, on three of these, the pore size distribution has been determined by means of Hg intrusion (Procedure 70-IMA-G-0-07 [82]).

The results obtained are presented in detail in the report 70-IMA-L-3-30 [83]. Table 5.4 shows the summary of the characteristics for the different types of blocks.

It can be observed that there is a greater density in the upper part of the blocks, although the deviations for the average are less than 0.04 g/cm³.

The average thermal conductivity in the direction perpendicular to the compaction force is always somewhat greater, contrary to what was observed in the laboratory tests (Section 4.4.3.).

The porosity study did not show variations between different positions within the same block nor between different types of blocks. The percentage of pores with radius greater than 3 μm is 23 ± 6, with a mode of 9 ± 4 μm; the percentage of pores with radius between 3 μm and 0.05 μm, is 26 ± 6, with a mode of 0.4 ± 0.3 μm; and the percentage of pores with radius less than 0.05 μm is 50 ± 10, with a mode about 0.008 ± 0.003 μm. Figure 5.6 shows the distribution of the pore sizes obtained in the upper, middle, and lower parts of the blocks sampled.

5.3. “In situ” test

The “in situ” test is being performed in the underground laboratory in Grimsel (Switzerland). For this test, a new drift was excavated, which is 70.40 m long and 2.28 m in diameter. In the last 17.00 m of this drift (the test zone), have been installed two heaters —each with a length of 4.54 m, an exterior diameter of 0.90 m, and separated by 1.00 m— inside a liner which is concentric with the drift. The test area of the drift is filled with blocks fabricated of highly-compacted bentonite (clay barrier), and sealed with a concrete plug. The test zone is shown, schematically, by one longitudinal and two cross sections in Figures 5.7 and 5.8.

Table 5.3
Characteristics of the blocks (*)

Block type	A	B+C (*)	D+E (*)
Thickness, in cm	12.25±0.08	12.25±0.06	12.24±0.14
R-r, in cm	31.50±0.00	30.60±0,00 (B)	30.60±0.00 (D)
Total density, in g/cm ³	2.02±0.02	1,99±0.02	2.06±0.04
Dry density, in g/cm ³	1.77±0.02	1.76±0.02	1.82±0.04
Water content, in %	14.2±1.4	13.2±0.9	13.1±0.9
Mass, in kg	25.8±0.3	25.2±0.2	25.3±0.4

(*)For the symbols, see Figure 5.3.

The activities of design and fabrication of the blocks for the construction of the clay barrier are described in detail in the report 70-AIT-L-3-10, *Informe final sobre las actividades realizadas por AITEMIN para la construcción de una barrera de bentonita* [84]. It should be noted that knowledge and experience have been gained from the “mock-up” test.

The fundamental activities were: design, of the blocks and the molds; fabrication, of the molds; fabrication, of the blocks; and packaging and storing of the blocks.

5.3.1. Design of the blocks and the molds

The following two specifications were written for the blocks and the molds: 70-AIT-H-3-01 *Specification for bentonite blocks* [85] and 70-AIT-H-3-02 *Specification of moulds* [86], respectively.

In the specifications for the bentonite blocks, the design data for the clay barrier, as geometry, dimensions, and nominal weights, as well as the dry density of the blocks ($1.70 \pm 0.02 \text{ g/cm}^3$) were taken into consideration. The nominal dry density of the blocks was determined taking into account

Table 5.4
Results of characterization of blocks

	Block type		
	A	B	C
N° of compacted blocks	598	322	92
N° of sampled blocks	8	5	2
Big-bag w (1), in %	12.6±0.3	14.4±1.5	
Dimension ρ _d (2), in g/cm ³	1.77±0.01	1.77±0.02	
Sections ρ _d (3), in g/cm ³	1.72±0.01	1.71±0.02	1.75
Sections w (3), in %	13.6±0.4	14.5±1.1	12.8
Sections S _r (3), in %	65	67	64
Upper ρ _d (4), in g/cm ³	1.74±0.01	1.73±0.01	1.77
Medium ρ _d (4), in g/cm ³	1.73±0.01	1.72±0.02	1.76
Lower ρ _d (4), in g/cm ³	1.70±0.01	1.67±0.06	1.74
Parallel thermal cond., in W/m · K	0.91±0.06	0.94±0.04	0.99
Perpendicular thermal cond., in W/m · K	0.96±0.05	0.95±0.07	1.02
Parallel thermal cond., in W/m · K (5)	0.88	0.92	0.87

(1) Water content of the clay from the big-bag with which the block was made.
(2) Calculated according to the block weight, the water content of the clay from the big-bag and the dimensions of the block measured after compaction.
(3) Average of the determinations made in fifteen samples taken in different positions inside the block.
(4) Average of the five determinations made in five samples distributed in the upper, medium and lower part of the block.
(5) According to the theoretical fitting determined for the FEBEX bentonite: $\lambda = 1.30 - 0.75 / (1 + \exp (S_r - 0.67) / 0.11)$.

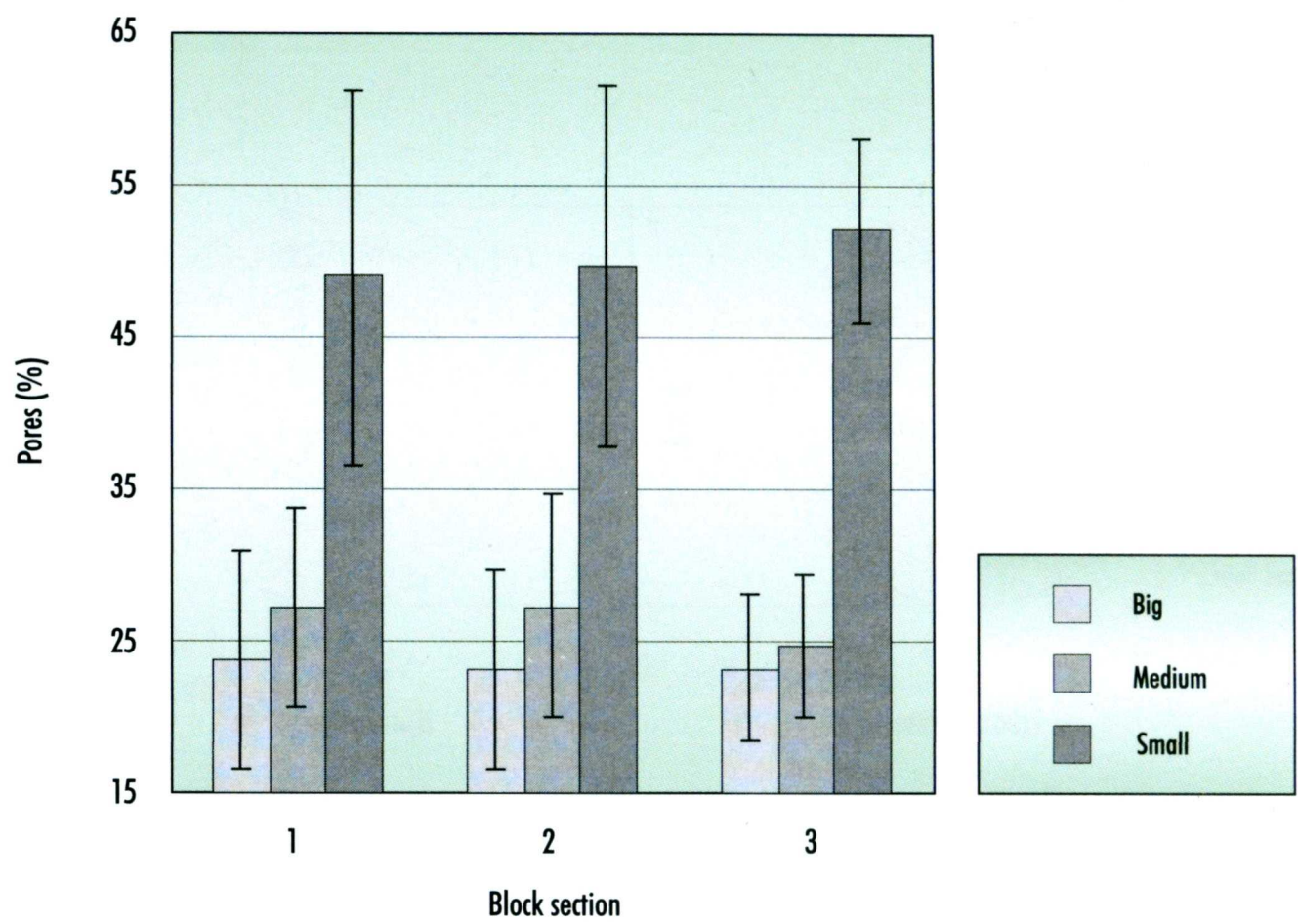


Figure 5.6: Pore size distribution in top, middle and bottom part of the sampled blocks.

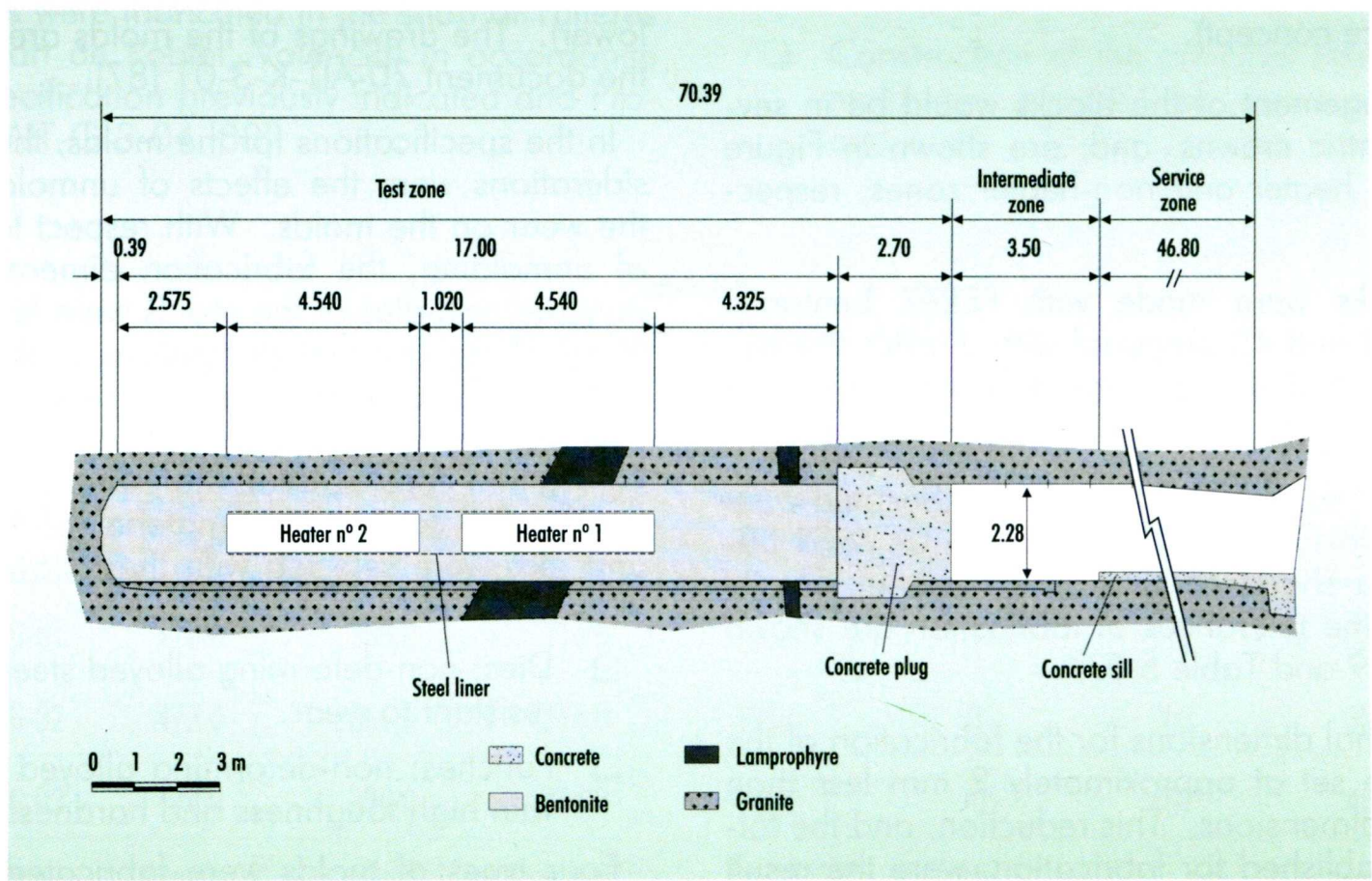


Figure 5.7: "In situ" test: longitudinal section.

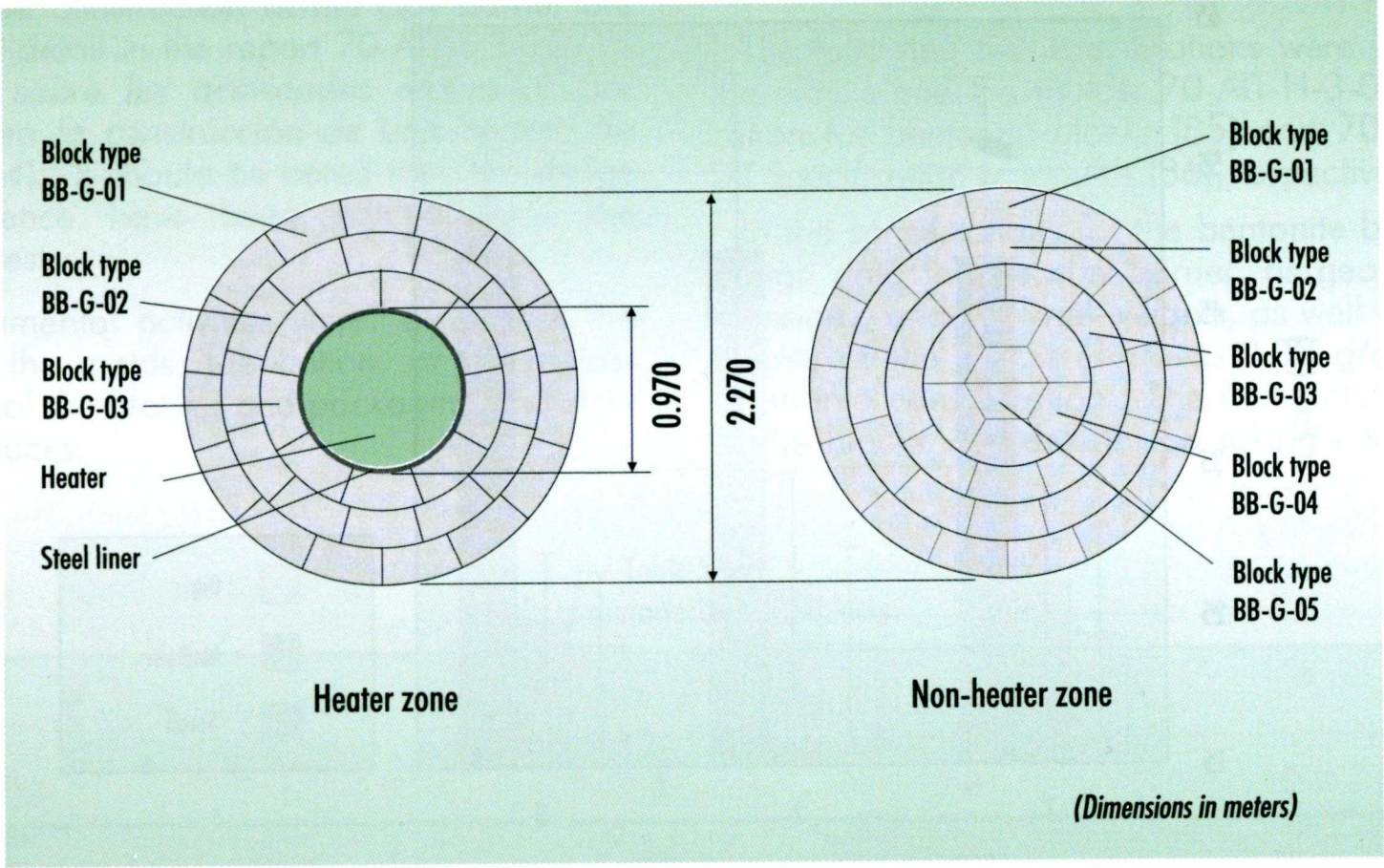


Figure 5.8: Arrangement of blocks in heater and non-heater zones.

the estimated volume of gaps as 7% and the fact that the final average dry density of the clay barrier was to be 1.60 g/cm³ (something less than that of the reference concept).

The arrangement of the blocks would be in several concentric crowns, and, are shown in Figure 5.8 for the heater and non-heater zones, respectively.

The blocks were made with FEBEX bentonite (Chapters 3 and 4), prepared with a grain size less than 5 mm, and with an average water content on the order of 14 %.

The five types of blocks that were fabricated were BB-G-01, BB-G-02, BB-G-03, BB-G-04, and BB-G-05. The shape of each, the nominal dimensions, and the tolerances of fabrication are shown in Figure 5.9 and Table 5.5.

The nominal dimensions for the fabrication of the blocks were set at approximately 2 mm less than the design dimensions. This reduction, and the tolerances established for fabrication, were the result of anticipating that the actual installation of the blocks in the test area would be easier with these conditions.

The most important elements of each one of the molds were: a die, two punches (an upper and a lower), and two punch holders (an upper and a lower). The drawings of the molds are included in the document 70-AIT-K-3-01 [87].

In the specifications for the molds, the main considerations were the effects of unmolding and of the wear on the molds. With respect to the effects of unmolding, the fabrication dimensions of the punches and dies of the molds were lessened from those of the nominal dimensions of the blocks by about 0.4%, to compensate for the possible expansion that the blocks might suffer upon unmolding. Taking into consideration the expected wear on the molds, the construction material of the punches and dies were selected with the following characteristics:

- ❑ Dies: non-deforming alloyed steel F521, very resistant to wear.
- ❑ Punches: non-deforming alloyed steel F522, with high toughness and hardness.

Four types of molds were fabricated: M-BB-01, M-BB-02, M-BB-03, and M-BB-04/05. The fabrication tolerances of the molds were fixed at ± 0.3 mm.

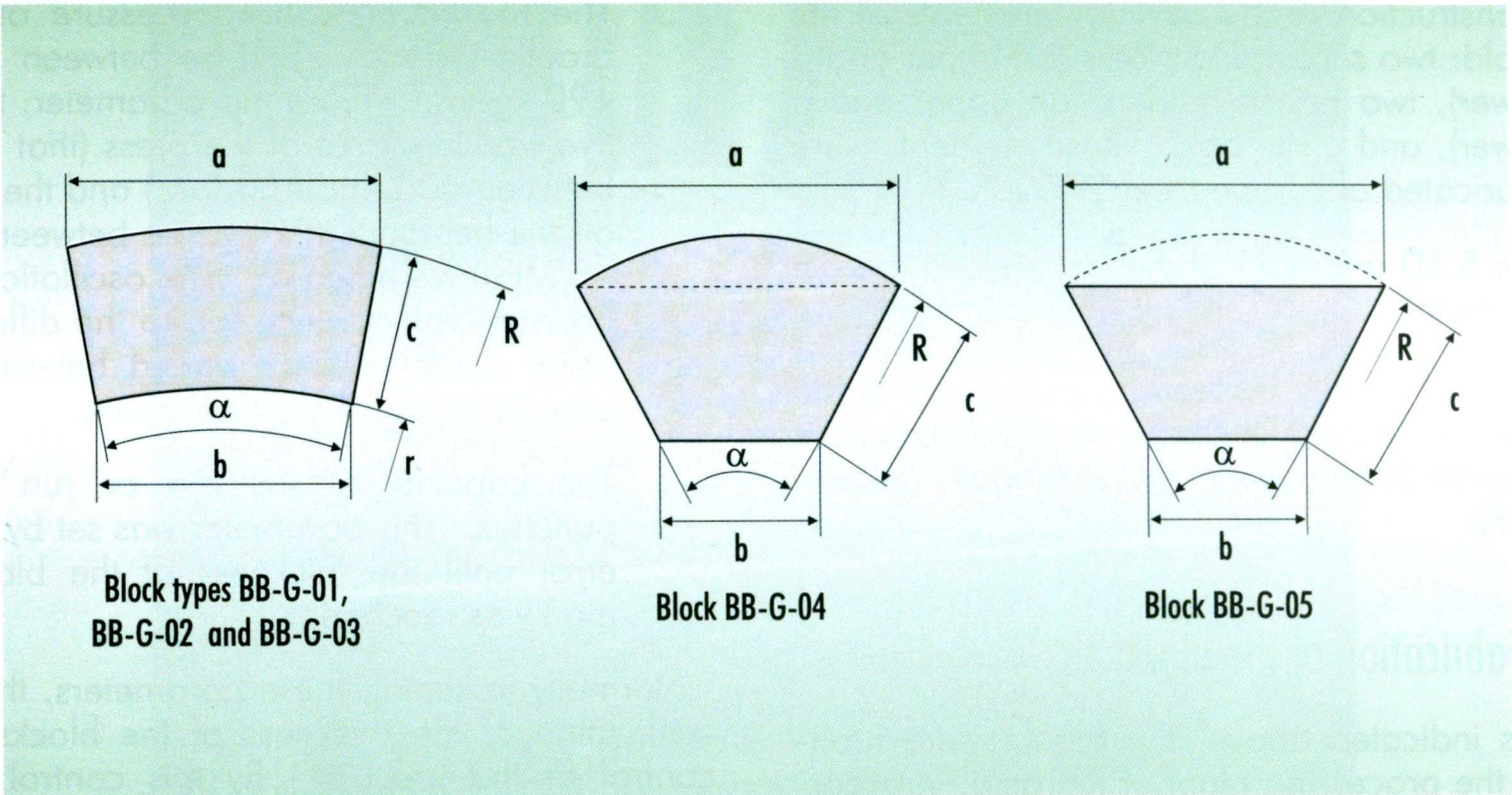


Figure 5.9: Shape and dimensions of the bentonite blocks

5.3.2. Fabrication of the molds

The molds were fabricated in the shop of Talleres Mateu (Quart de Poblet, Valencia) in accordance with the specification previously indicated and Procedure 70-AIT-G-3-04 [88].

Fundamentally, the fabrication of the molds consisted of the following activities:

- Construction of the principal elements of the mold: two punches (an upper and a lower) and a die. These elements were fabricated

Table 5.5
Shape and dimensions of the bentonite blocks

Block type	Surface dimensions mm			Thickness mm	Radius mm		Nominal angle α
	a	b	c		R	r	
BB-G-01	470.0 ^{+2,0} _{-5,0}	380.0 ^{+2,0} _{-4,0}	214.0 ^{+2,0} _{-3,0}	125.0 ^{+2,0} _{-2,0}	1 133	919	24°
BB-G-02	473.0 ^{+2,0} _{-5,0}	361.0 ^{+2,0} _{-4,0}	214.0 ^{+2,0} _{-3,0}	125.0 ^{+2,0} _{-2,0}	917	703	30°
BB-G-03	478.0 ^{+2,0} _{-5,0}	330.0 ^{+2,0} _{-3,0}	214.0 ^{+2,0} _{-3,0}	125.0 ^{+2,0} _{-2,0}	701	487	40°
BB-G-04	483.0 ^{+2,0} _{-5,0}	240.0 ^{+2,0} _{-3,0}	240.0 ^{+2,0} _{-3,0}	125.0 ^{+2,0} _{-2,0}	485	-	60°
BB-G-05	483.0 ^{+2,0} _{-5,0}	240.0 ^{+2,0} _{-3,0}	240.0 ^{+2,0} _{-3,0}	125.0 ^{+2,0} _{-2,0}	-	-	60°

of alloyed steel for tools F521 (die) and F522 (punches).

- Construction of the auxiliary elements of the mold: two supporting plates (an upper and a lower), two punch holders (an upper and a lower), and a die box. These elements were fabricated of carbon steel ST-52.

Figures 5.10 and 5.11 show the die and the die box of a mold.

The logs of quality inspection, made during the mold fabrication, are included in the *Dossier de calidad sobre la fabricación de los bloques de bentonita* [89].

5.3.3. Fabrication of the blocks

As was indicated above, the blocks were fabricated in the processing plant of Refracta, in accordance with the specifications previously indicated and Procedure 70-AIT-G-3-01 [90].

5.3.3.1. Fabrication process

The blocks were fabricated with an uniaxial double-acting hydraulic press with a maximum effective force of 550 tons, following the operations described below.

Material preparation

This operation consisted of mixing the bulk bentonite in a machine for 15 minutes. After the mixing, a sample was taken to determine, approximately, the water content of the bentonite in the big-bag, with the object of equalizing the water contents, and, if it was necessary, mixing the bentonite of various bags. The operation was made for each 500 kg of bentonite.

Selection of the press working conditions

This operation was made at the beginning of the fabrication of each type of block and each day of work. During the compaction of the blocks, the conditions of the work were constantly checked. The checking consisted, fundamentally, of the press operator controlling the thickness of the blocks.

The working conditions were established using the following parameters:

- The maximum working pressure of the hydraulic system was to be between 170 and 190 kg/cm². From this parameter, the effective working force of the press (that oscillates between 400 and 450 tons) and the pressure on the bentonite (that varies between 40 and 45 MPa) were known. The oscillations of the pressure values were due to the difference in water content, which varied between 12 % and 16 %, of the bulk bentonite.
- The capacity of the die or run between punches. This parameter was set by trial and error until the thickness of the block (125 mm) was reached.

Normally in setting these parameters, the simple verification of the thickness of the blocks permits control of the process. By this control, the following incidents were detected:

- Higher water content than expected in the bulk material, which was manifested by a smaller thickness than for the normal set of working conditions.
- Loss of mass of the material which was manifested by a smaller thickness.
- Heating of the oil in the hydraulic system of the press that was manifested by arbitrary changes in the thickness. When this happened, the work was interrupted for several hours or for the rest of the working day.

In these three cases, as in any other, remedial action was taken whenever the thickness of a block differed from the nominal by more than ± 1 mm. Thus, the compaction process was always under control.

Block formation

This operation consisted in weighing the mass of bentonite stipulated for each type of block; introducing it into the die; mechanically compacting and ejecting the block; manually retrieving the block; and checking the block, by the press operator (Figure 5.12).

The nominal weight of each of the different types of bentonite blocks was: 22.1 kg for BB-G-01; 21.8 kg for BB-G-02; 21.3 for BB-G-03; and 23.1 kg for BB-G-04/05.



Figure 5.10: View of die of the mold. Over the die are situated two punches of the mold.

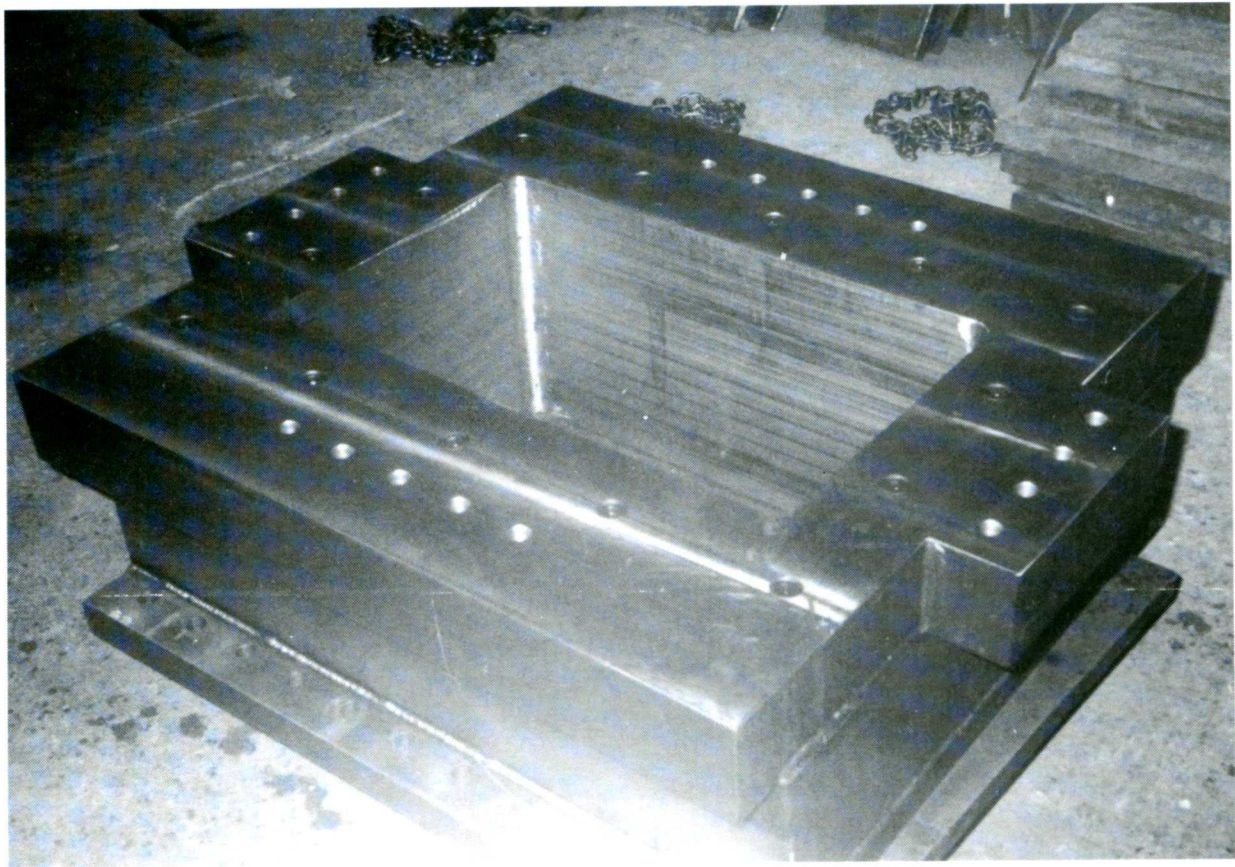


Figure 5.11: View of die box of the mold.

With these activities, the fabrication of the blocks BB-G-01, BB-G-02, BB-G-03, and BB-G-04 were completed (Figures 5.13 and 5.14). Changes to the shape of BB-G-04 were made to obtain block type BB-G-05, by using a cutting machine to remove a cylindrical segment (Figure 5.15) with a diamond disk (800 mm in diameter and 7.5 mm in thickness). The cut was made perpendicular to the two faces of the block.

5.3.3.2. Quality control and physical characteristics
(dry density and water content)
of the bentonite blocks

Table 5.6 shows the number and destination of each type of block fabricated. The majority of the blocks were sent to Grimsel; however, some were

sent to AITEMIN, in Toledo, to make a trial installation.

The inspection methods and tests on the blocks are described in the following five procedures: 70-AIT-G-3-02 [91]; 70-AIT-G-3-05 [92]; 70-AIT-G-3-06 [93]; 70-AIT-G-3-07 [94]; and 70-AIT-G-3-08 [95].

Procedure 70-AIT-G-3-02 [91] should be especially noted. The control of the blocks was based on the following sequential inspections: a single inspection, that was made at the beginning of the fabrication process of each type of block by the fabricator; a total inspection, that was made on the thickness and external aspect of the blocks by the fabricator; and a double sampling inspection made by both the fabricator of the blocks and by AITEMIN.



Figure 5.12: Frontal view of the mold installed in the press. In this photo the upper punch is in stand position and the lower punch (not shown) is at the high of the upper face of the die (press table), supporting the bentonite block which has just been compacted.

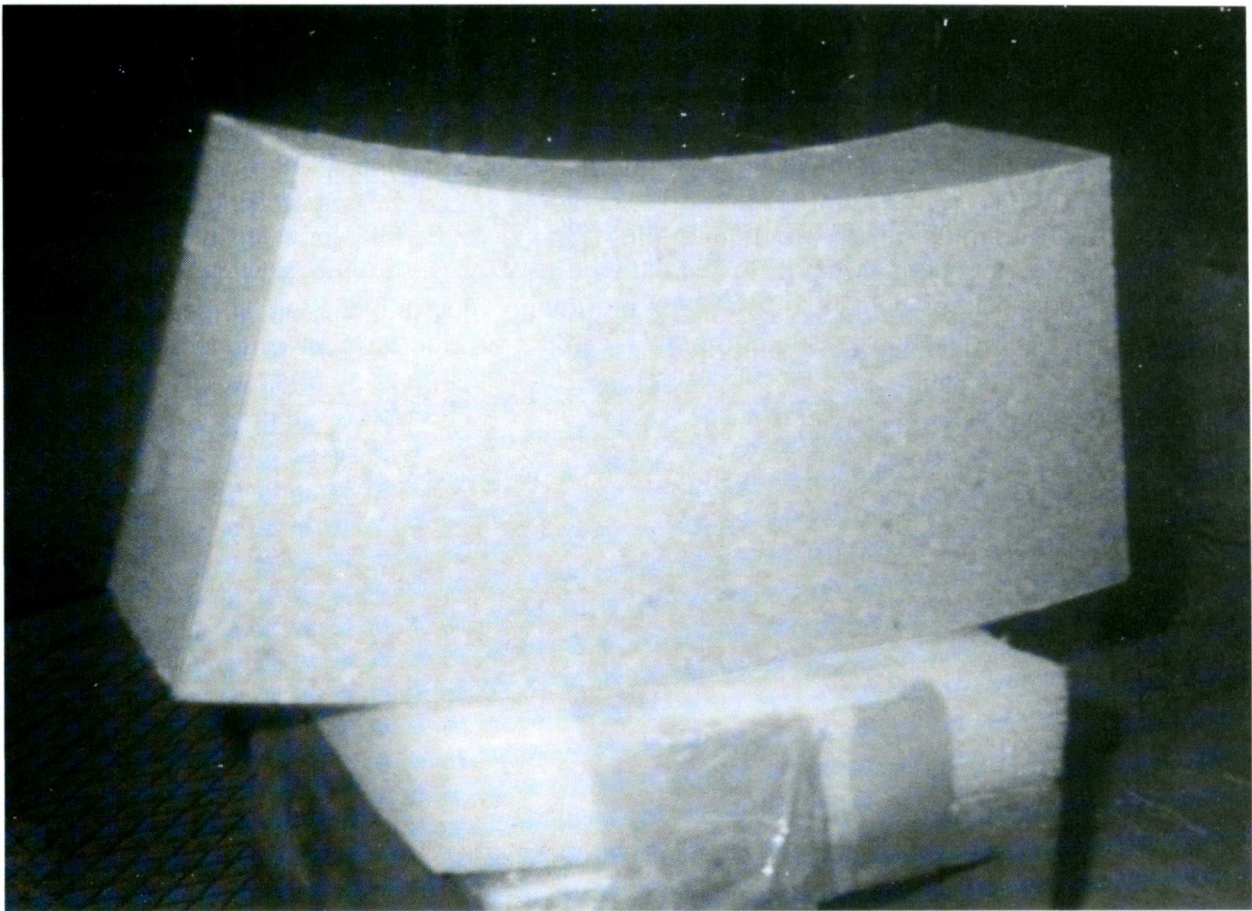


Figure 5.13: Close-up of fabricated block (BB-G-01).

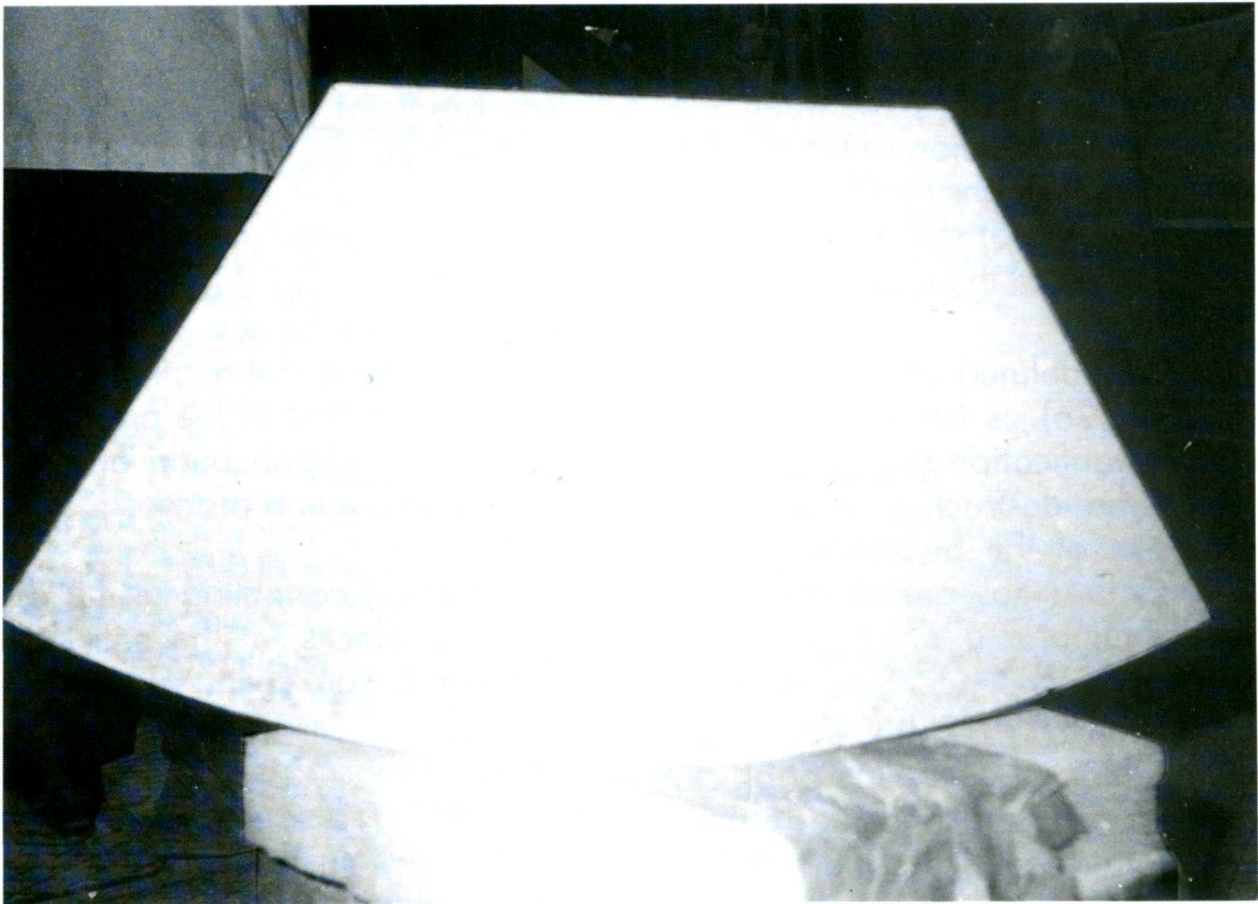
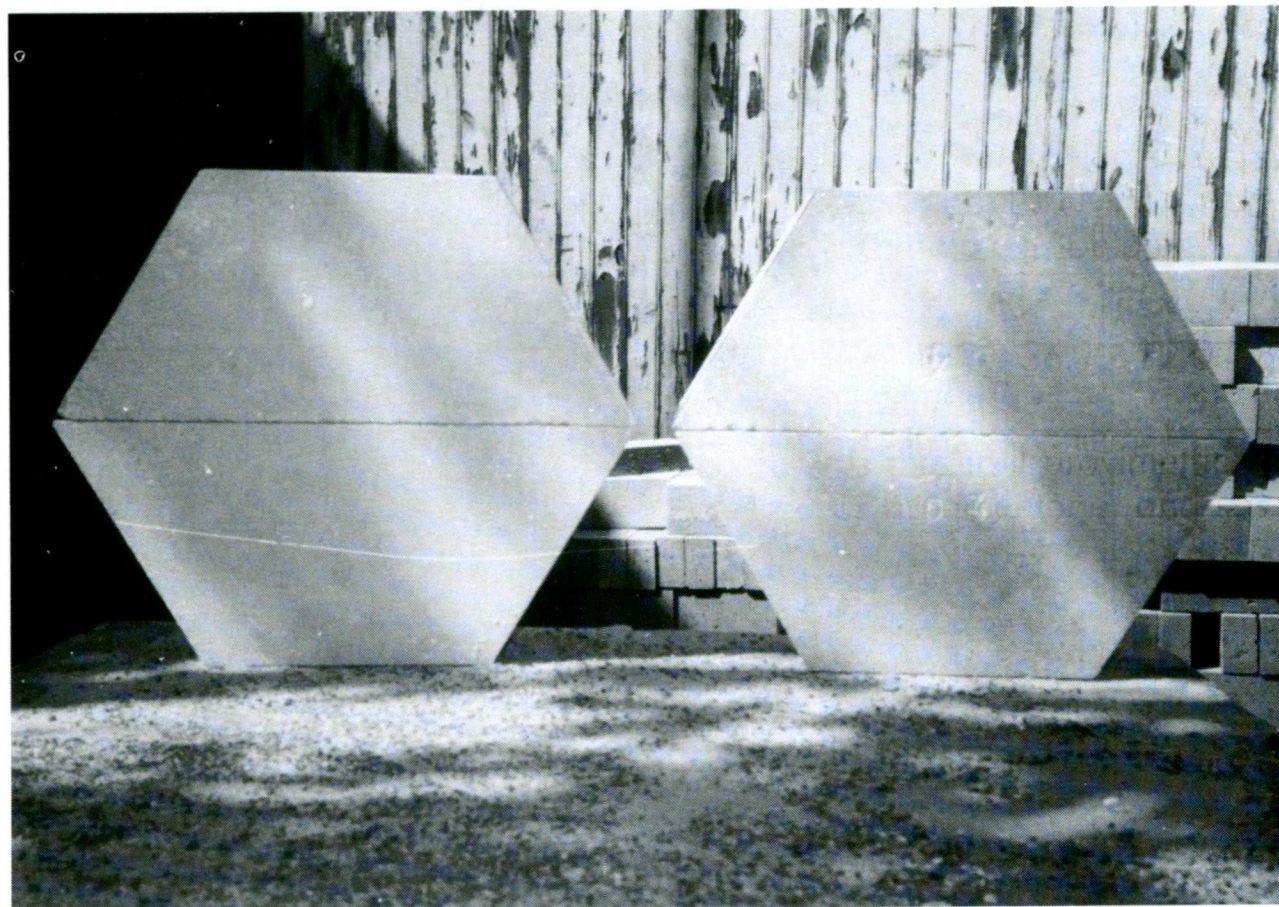


Figure 5.14: Close-up of fabricated block (BB-G-04).



*Figure 5.15: Close-up of four of type BB-G-05, after being mechanized, as they will be installed.
Note: The two cut surfaces of the blocks are placed together.*

In respect to the control methods of the fabrication processes, the following controls are specially noted: selection of the working conditions of the press (effective force and difference between the runs of the pistons), in the fabrication of the blocks; and the characteristics of the construction material of the punches and dies (type of steel and thermal treatment), in the fabrication of the molds.

The sampling inspection defined according to the standard ISO 5022/79 [96], is based on the results of non-destructive verification (external aspect and dimensions) and the destructive tests (water content and dry density) of the bentonite blocks. The defects or non-conformities are classified as major, intermediate, or minor.

The sampling inspection by AITEMIN was made at the end of the fabrication of each type of block. The number of samples analyzed and the inspection lot (15 % of the total blocks) are indicated in Table 5.7.

First the non-destructive tests were made: control of the dimensions and of the external aspect; then

the destructive tests: determinations of water content and dry density.

The results of the non-destructive tests are shown in Table 5.8.

The irregularities encountered consisted of small surficial fissures in the faces and in the rounded edges of some blocks. The first were detected at the beginning of fabrication and the solution was to reduce the flow of the press; the second were at the end of the fabrication and the solution was a second compaction process.

Some small crumbling at the edges and corners of the cut blocks, at the entrance and exit of the cutting disk, were seen.

These minor irregularities or non-conformities appeared in a few of the blocks that were analyzed and were within the specification limits of the inspection plan.

The results of the destructive tests are presented in Table 5.9.

Table 5.6:
Number of fabricated blocks

Type Unit weight (kg)		BB-G-01 22.1	BB-G-02 21.8	BB-G-03 21.3	BB-G-04 23.1	BB-G-05 18.0
Grimsel	Units	2 520	1 974	1 483	486	162
	Weight (kg)	55 692	43 034	31 588	11 227	2 916
Toledo	Units	360	288	144	48	16
	Weight (kg)	7 956	6 279	3 068	1 109	288
Total	Units	2 880	2 262	1 627	534	178
	Weight (kg)	63 648	49 312	34 655	12 335	3 204

Table 5.7
Inspection lots and samples made by AITEMIN

Type		BB-G-01	BB-G-02	BB-G-03	BB-G-04	BB-G-05
Number of blocks	Units	2 880	2 262	1 627	534	178
	Weight (t)	63.65	49.31	34.66	12.33	3.20
Inspection lot	Units	432	340	244	80	27
	Weight (t)	9.55	7.40	5.20	1.85	0.48
Inspection sample	Units	50	50	32	13	13
	N° of END	50	50	32	13	13
	N° of ED	9	9	9	6	6

t = Ton; END = Non-destructive tests; ED = Destructive tests.

Table 5.8
Results of non-destructive tests made by AITEMIN

Type	BB-G-01	BB-G-02	BB-G-03	BB-G-04	BB-G-05
External aspect	R (46) I (4)	R (50)	R (32)	R (13)	R (10) I (3)
Regularity of surfaces and sides	R (50)	R (50)	R (32)	R (13)	R (13)
Dimensions	DT (50)	DT (50)	DT (32)	DT (13)	DT (13)

R = Regular; I = Irregular; DT = Within tolerances; (50) Number of units.

As can be observed in the table, the average values of the water content, as well as the dry density, are within the established specification limits for the design of the blocks: 1.68 g/cm³ to 1.72 g/cm³, for the dry density and 11% to 15%, for the water content.

The quality inspection logs made for the fabrication process and on the bentonite blocks, are included in *Dossier de calidad sobre la fabricación de los bloques de bentonita* [89].

5.3.4. Packaging and storing of the blocks

The packaging and storing of the blocks in the processing plant were done in accordance with Procedure 70-AIT-G-3-03 [97]. The number of packages that were needed for the distinct types of blocks are indicated in Table 5.10.

The blocks were packaged in cardboard boxes placed on wooden pallets. Inside the box was

placed a plastic bag and the bottom was layered with plastic sponge, over which were placed the blocks in 6 layers of 7 blocks each (except for the block BB-G-04, with 6 blocks per layer); the final layer was covered with a layer of the same plastic sponge material. Between each layer and between the blocks of the same layer, separating strips were placed and fillers were used between blocks.

After the box was closed, tags, protectors for the edges, packaging strips, and plastic bags were placed on the exterior, then the filled package was placed in a thermal chamber to hermetically seal the plastic bag over the entire box, i.e. shrink-wrapped.

In Figures 5.16 and 5.17 are shown photos of the packaging process and the aspect of the final package.

The quality inspection logs on the handling, packaging, and storage of the blocks, are included in the *Dossier de calidad sobre la fabricación de los bloques de bentonita* [89].

Table 5.9
Results of the destructive tests made by AITEMIN

Sample	BB-G-01		BB-G-02		BB-G-03		BB-G-04		BB-G-05	
	ρ _d	w	ρ _d	w	ρ _d	w	ρ _d	w	ρ _d	w
1	1.67	13.9	1.69	13.4	1.71	14.7	1.69	15.3	1.71	13.1
2	1.70	14.1	1.70	14.4	1.69	15.3	1.72	13.0	1.70	13.5
3	1.70	14.9	1.69	14.7	1.70	15.0	1.70	12.7	1.69	13.1
4	1.69	14.9	1.70	13.8	1.68	14.3	1.71	13.2	1.72	12.9
5	1.71	13.8	1.69	14.2	1.70	14.8	1.68	15.3	1.72	12.8
6	1.68	15.0	1.68	15.4	1.69	14.1	1.68	12.6	1.71	13.1
7	1.70	14.3	1.69	13.2	1.68	15.3	-	-	-	-
8	1.69	14.1	1.69	12.3	1.68	15.6	-	-	-	-
9	1.70	15.4	1.68	15.3	1.69	14.8	-	-	-	-
ARITHMETIC AVERAGE	1.69	14.45	1.69	14.1	1.69	14.9	1.70	13.7	1.70	13.1

Table 5.10
Packages utilized for the distinct types of blocks

Type		BB-G-01	BB-G-02	BB-G-03	BB-G-04	BB-G-05
N° of blocks in a full package		42	42	42	36	42
Mass weight for a full package (kg)		980	950	950	880	810
N° of packages	Grimsel	60	47	36	14	4
	Toledo	9	7	4	1	1



Figure 5.16: Photo of packaging of the blocks



Figure 5.17: Finished package of blocks. Shown are the base of package (pallet), the reinforced corners, the labeling, and the shrink wrap.

6

CONCLUSIONS

6.1. Origin and general aspects of the febex bentonite

The FEBEX bentonite has been extracted from the Cortijo de Archidona, developed by Minas de Gádor, S. A. , in the zone of Serrata de Níjar (Province of Almería, Spain). This deposit has been selected, in the R&D plans of ENRESA prior to the FEBEX project, as the most suitable material for backfilling and sealing a HLW repository, for its very high content of montmorillonite, large swelling pressure, low permeability, acceptable thermal conductivity, good adsorption properties, and ease of compaction for fabrication of blocks.

After selection of this deposit as the reference bentonite (called sample or bentonite S-2 in reports and publications), there have been numerous characterization and behavior (thermal, hydraulic, mechanical, and geochemical) studies of this bentonite. Consequently, there exists an extensive database of its properties. These data were used in the preliminary modelizations for the design of the two large-scale tests of the FEBEX project.

Some 300 tons of bentonite, suitably homogenized and conditioned, were acquired for FEBEX. Based on the prior studies, the selected bentonite needed to meet the following specifications:

- ❑ Fraction of particles greater than 5 mm, less than 5%, and fraction of particles less than 74 μm , more than 85%.
- ❑ Liquid limit greater than 90%.
- ❑ Swelling pressure between 3.0 MPa and 7.0 MPa, for a dry density of 1.60 g/cm^3 .
- ❑ Water content between 12.5% and 15.5%, after conditioning.

A bentonite deposit, even though very homogeneous, has horizontal as well as vertical variations. For a project such as FEBEX, it is important to use material as homogeneous as possible and to determine its properties by specific tests on samples of the same material so as to reduce the uncertainties in the subsequent modelizations and the final interpretation of the results of the entire experiment. For a performance assessment (PA) of a repository, nevertheless, it is necessary to know the range in the variations of the relevant properties of

a massive source of supply of bentonite. Thus, it is important to compare the properties of the bentonites S-2 and FEBEX, which were obtained from the same deposit but in two development phases (levels), more than five years apart.

Since, during this pre-operational stage, the time has been too short (only a few months) to make a complete characterization of the FEBEX bentonite, it has been necessary, for modelling, to use some properties obtained for the bentonite S-2.

The comparison of the results of the two available sets of characterization tests —the bentonite S-2 and the FEBEX bentonite— indicates that the deposit is very homogeneous, which legitimizes the use of certain parameters from the tests on S-2 for the pre-operational stage modelizations. However, there are some differences between these two sets, which permit an evaluation of the range of variations in the properties. This evaluation can be utilized to establish limits on the modelling sensitivity analysis and thus gain an initial idea of the consequences in the performance assessment of a repository. These differences also indicate the necessity of a detailed characterization of the FEBEX bentonite during the operational stage (three or four years), in order to reduce the uncertainties in the final evaluation of the global results of this experiment.

In the following sections some general conclusions about the relevant properties of the bentonite from this deposit are presented and the operational stage testing program is outlined.

6.2. Mineralogical and chemical composition

Mineralogical composition

The bentonite from the Cortijo de Archidona deposit has a very high content of dioctahedric smectite of the montmorillonite type; furthermore, from the mineralogical point of view, the deposit is very homogeneous: the montmorillonite content of both bentonites (S-2 and FEBEX) varies between 88% and 96%. Some of the accessory minerals include quartz, plagioclase, potassium feldspar, calcite, and tridymite.

The analyses of CSIC-Zaidín on the FEBEX bentonite detected interstratified smectite/illite with an estimated content of some 15%, whereas this had not been detected in the bentonite S-2. The influence that the interstratified smectite/illite can have in the physico-chemical behavior of the bentonite has not been determined in this pre-operational stage.

Chemical composition

Table 6.1 shows the values of some of the major chemical elements of the bentonites S-2 and FEBEX.

The content of the major elements, obtained in the two laboratories, is very similar for both bentonites. There are, however, relatively large differences between the two laboratories for some elements: K_2O for the bentonite S-2; Na_2O and Fe_2O_3 for the FEBEX bentonite. Neither the reasons for these differences nor their effects on the physico-chemical behavior have been analyzed.

The FEBEX bentonite shows a lower salinity than that of the S-2. The lower salinity is due, basically, to the lower concentration of the chloride ion.

Table 6.2 shows the average values of the exchangeable cation content. It also shows the cation exchange capacity (CEC).

The values for the cation content obtained by the two laboratories are practically equal for the FEBEX bentonite; however, the CEC does differ as different methods for the determinations have been employed (Section 4.3.5.).

There are relatively large differences not only between the results of the two laboratories for the bentonite S-2, but also between the two bentonites, chiefly in the CSIC-Zaidín results; in addition, the standard deviations of the two laboratories are greater for the results from the S-2 than from the FEBEX, especially in the values of the CEC. In any case, it seems that the CEC is lower for the bentonite S-2 than for the FEBEX, which may imply differences in the physico-chemical behavior.

6.3. Identification properties

The data that are presented in Table 6.3 are being called the identification properties (following the geotechnical terminology) because they give an idea, by means of simple tests, of the type of physico-chemical behavior of a clay.

The value of the liquid limit of the two bentonites, S-2 and FEBEX, is very similar in the CIEMAT tests. That obtained by UPC-DIT for the FEBEX bentonite is somewhat lower. Nevertheless, whichever value

Table 6.1
Chemical composition of the bentonites S-2 and FEBEX

Oxide %	Bentonite S-2		FEBEX bentonite	
	CSIC-Zaidín ⁽¹⁾	CIEMAT ⁽²⁾	CSIC-Zaidín ⁽³⁾	CIEMAT ⁽³⁾
SiO ₂	59.9	57.3	58.9	59.4
Al ₂ O ₃	18.7	19.6	19.5	18.1
Fe ₂ O ₃	3.6	3.9	3.5	2.9
MgO	5.8	4.7	4.8	4.2
CaO	2.0	2.9	2.5	1.9
Na ₂ O	2.2	1.9	2.3	1.4
K ₂ O	0.7	1.4	1.2	1.1

(1) Average values of various determinations (2) Values of only one determination (3) Average values of four determinations

Table 6.2
Average values of the cation exchange complex

Cations and CEC meq/100 g	Bentonite S-2		FEBEX bentonite	
	CSIC-Zaidín	CIEMAT	CSIC-Zaidín	CIEMAT
Ca ²⁺	31 ± 6	41 ± 7	47 ± 2	48 ± 4
Mg ²⁺	33 ± 6	31 ± 7	36 ± 2	38 ± 2
Na ⁺	30 ± 3	22 ± 3	26 ± 1	25 ± 1
K ⁺	3 ± 1	2.5 ± 0.4	2.3 ± 0.1	2.2 ± 0.0
CEC	92 ± 12	97 ± 17	106 ± 3	113 ± 6

Table 6.3
Identification properties

Property	Bentonite S-2		FEBEX bentonite		
	CSIC-Zaidín	CIEMAT	CIEMAT	UPC-DIT	CSIC-Zaidín
Water content in equilibrium with laboratory conditions, in %		10 to 13	12 to 15		
Liquid limit, in %		105 ± 10	103 ± 4	93 ± 1	
Specific weight		2.78	2.70 ± 0.05		
Grain-size distribution, in %					
Fraction less than 74 µm	93 ± 3	86	93 ± 1	87	
Fraction less than 2 µm	82 ± 6	65 ± 1	65 ± 3	45	
Specific surface, in m ² /g					
Total	614 ± 74	516 ± 37			640 a 659
External, BET		37	32 ± 3		

is considered, it is too low for that of a bentonite with such a high content of montmorillonite and that has a relatively high content of sodium as an exchangeable cation. The liquid limit is an index of the mechanical behavior of a clay, and a low liquid limit in relation to the mineralogy signifies, in principle, that the physical behavior of this bentonite does not correspond to its mineralogical characteristics.

Regardless of the differences in the grain-size distributions between the bentonites S-2 and FEBEX, it should be noted that there is a large difference between the results of CIEMAT and UPC-DIT in the content of the fraction less than 2 µm (clay size). The proportion of the clay size fraction depends on the pre-test treatment of the bentonite before the grain-size determination. The differences are explained by the fact that CIEMAT made a very

strong dispersion, including using ultrasound, whereas UPC-DIT employed a standard geotechnical technique. Or, in other words, the results of such a simple determination could also be an index of physico-chemical behavior.

The average values of the total specific surface, which are very similar for the two bentonites, are somewhat lower than that for a bentonite with as much montmorillonite content. In fact, compared with a typical montmorillonite, it seems that the montmorillonite content in the bentonite of the Cortijo de Archidona deposit would be something lower to that estimated in the mineralogical analyses, confirming the existence of a certain percentage of interstratified smectite/illite [44]. Also the value of the external specific surface (BET) is low comparing to the usual values for smectites found in the scientific literature.

The low content of the fraction less than 2 μm was noted in the first studies made on the bentonite S-2 [9] and an explanation was proposed that the smaller particles have become agglutinated or cemented with colloidal silica (during the devitrification of the volcanic material), which would make it difficult to disperse the clay and, consequently, limit the separation of the fraction less than 2 μm . This argument is supported by the high content of phyllosilicates (62 %) found in the fraction greater than 20 μm [13]. Another explanation is that the greater part of the material of silt size, and some of the sand size, are pseudomorphs of smectite that keep the size and the shape of the grains of the volcanic rock from which they come; the pseudomorphs are relatively stable and would moderate the physical behavior of the bentonite: they chemically act as smectite but do not have the physical effects of the bentonite [98].

In any case, it seems that the fact that exists a strong agglomeration of the fine particles explains the low values of the liquid limit and the external specific surface.

In this stage of the study, the influence that the peculiarities described can have in the physico-chemical behavior of the FEBEX bentonite was not investigated. However, it has been found that, with several cycles of heating (between 150°C and 200°C) and cooling, the liquid limit increases from 98% to more than 190%.

6.4. Swelling pressure

For the swelling pressure of the bentonite S-2, a regression curve as a function of the dry density was determined. This curve is presented in Figure 6.1 and is expressed by the equation (Section 4.4.1.)

$$P_s = \exp(5.9 \rho_d - 7.9)$$

where P_s is the swelling pressure in MPa and ρ_d is the dry density in g/cm^3 .

Two set of tests, each with nominal dry density of either 1.60 g/cm^3 or 1.70 g/cm^3 , have been made for the FEBEX bentonite. The average values determined were slightly higher than those corresponding to the bentonite S-2 obtained when the above formula is applied: on the order of 7% and 10% for the nominal dry densities of 1.60 g/cm^3 and 1.70 g/cm^3 , respectively.

It is possible that there really exists a difference in the swelling pressures. However, for all practical purposes in the performance assessment of a repository, it can be considered that the difference is negligible.

6.5. Suction/water content relationship

For the bentonite S-2, for suctions between 2.0 MPa and 385.0 MPa, the following empirical equation (Section 2.5.10.) was determined

$$w = 36.1 - 12.0 \log s \quad r^2 = 0.94 \quad 191 \text{ points}$$

where, for this equation and the following one, w is the final gravimetric water content, expressed in percent, and s is the suction in MPa.

Likewise, for the FEBEX bentonite, UPC-DIT obtained the equation (Section 4.4.2.)

$$w = 40.7 - 14.3 \log s$$

CIEMAT also made suction versus water content tests for the FEBEX bentonite, but only for three values of suction, which did not permit the establishment of a mathematical relationship.

Figure 6.2 shows the regression curves for both bentonites and the points obtained by CIEMAT for the S-2 and FEBEX bentonites.

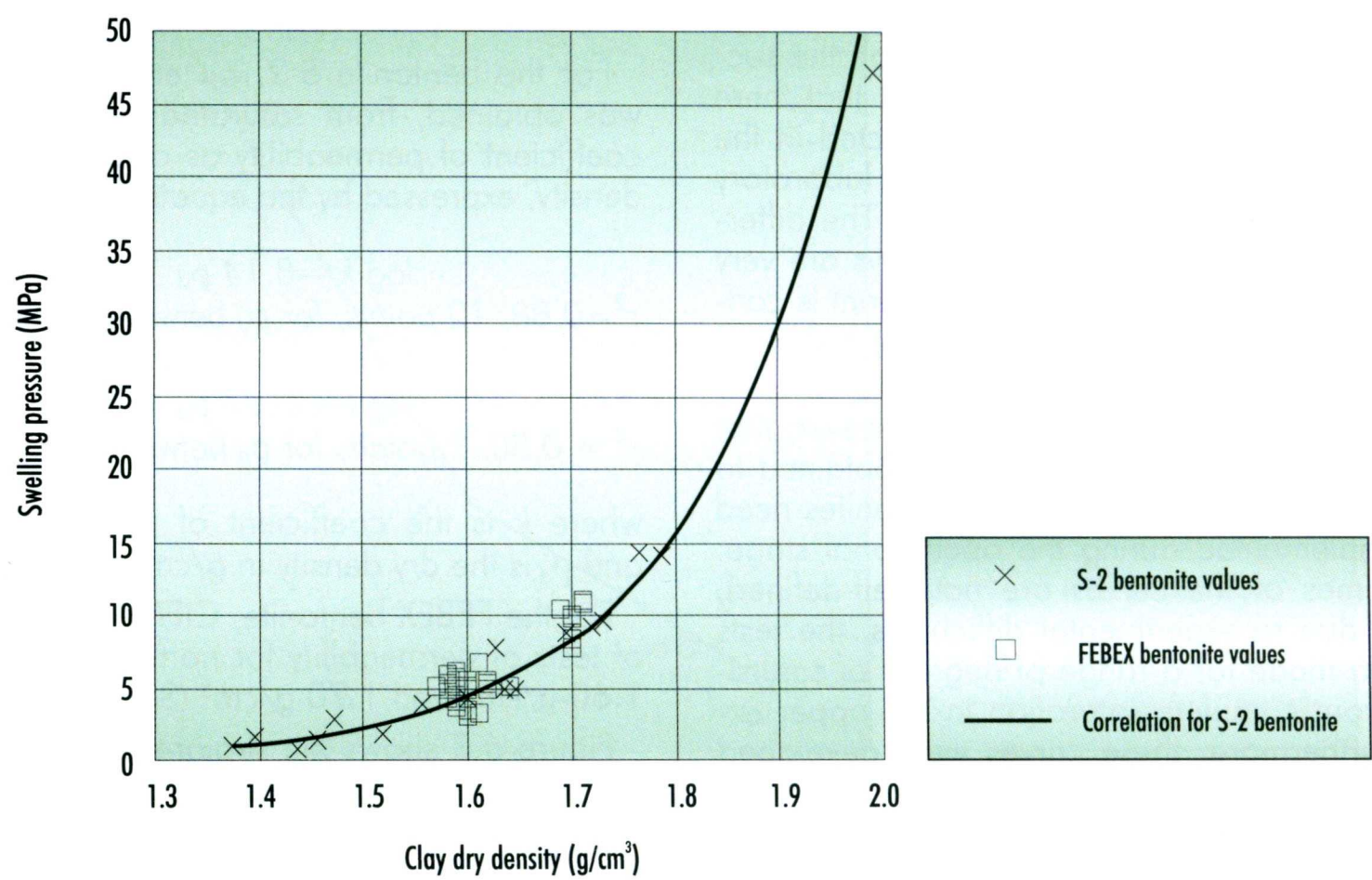


Figure 6.1: Swelling pressure vs. dry density.

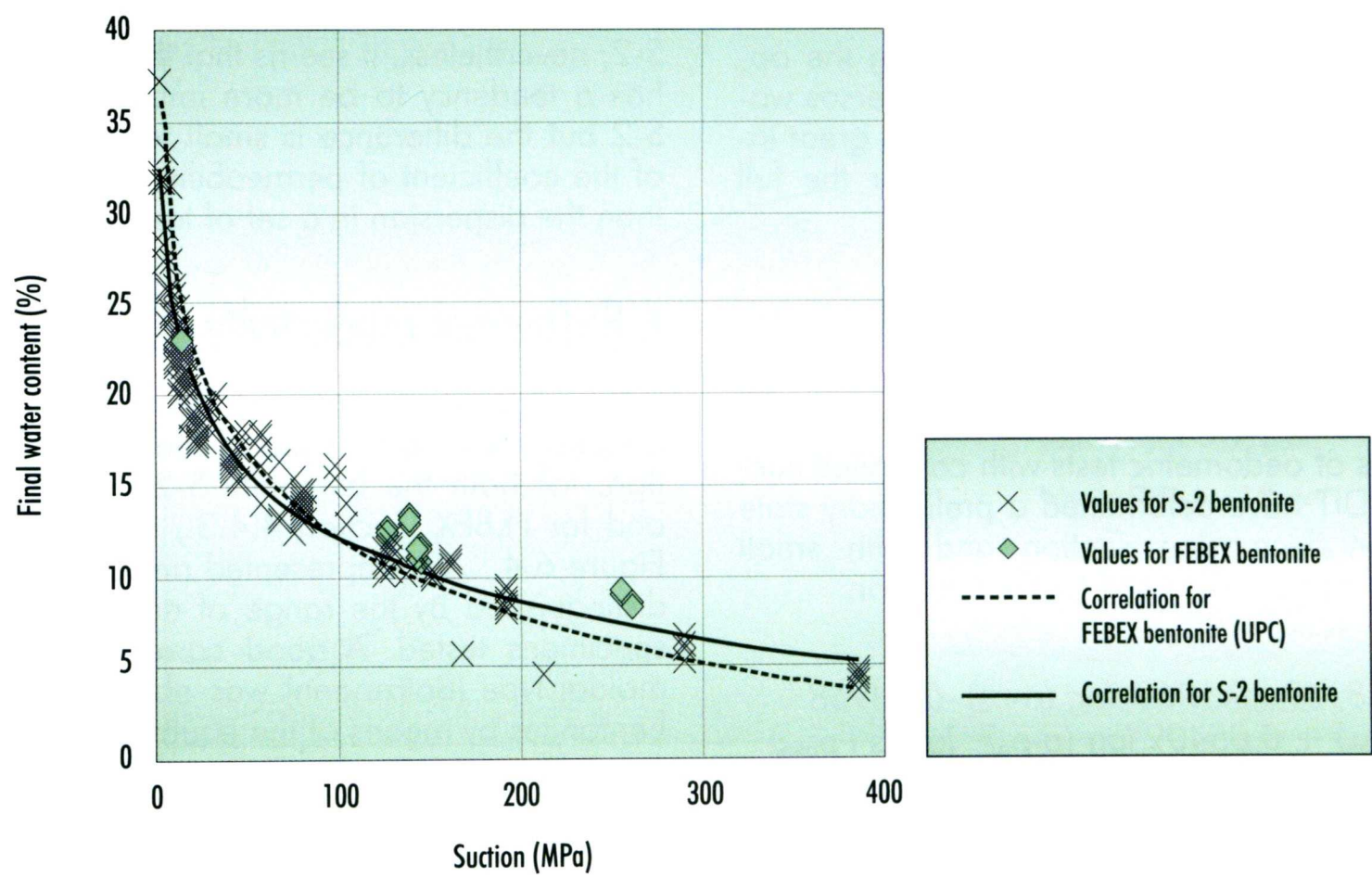


Figure 6.2: Suction/water content relationship.

As can be observed, the differences between the two curves are very small, one or two percentage points in the water content, depending on the suction value. Nevertheless, it can be said that there is a certain difference, that is also reflected in the water content in equilibrium with the laboratory conditions, as indicated in Table 6.3. The differences, in any case, between the two clays are very small as far as the performance assessment is concerned.

It is necessary to take into account, however, that the suction/water content relationships obtained to date in the sets of tests for the two bentonites need to be supplemented during the operational stage. The extremes of the curves are not well-defined, because, due to experimental difficulties, the tests have been made for a range of degrees of saturation far from complete saturation in the upper extreme. Furthermore, these curves were developed from tests on different specimens and thus are not characteristic suction/water content curves developed from a single specimen. A characteristic suction/water content curve for a wetting path differs from one obtained for a drying path and this aspect has not been analyzed.

Also, to date, the influence of the temperature in the suction/water content relationship has not been defined with sufficient reliability.

In summary, it will be necessary, during the operational stage, to complete the suction versus water content study as this relationship has a great influence in the prediction of the time for the full saturation of the clay barrier.

6.6. State surface

By means of oedometric tests with controlled suction, UPC-DIT have determined a preliminary state surface for decreasing suction and with small changes of stress, expressed by the equation

$$e = 0.8169 - 0.1268 \log(\sigma - p_a) - 0.04968 \log(s + p_{atm}) + 0.05409 \log(\sigma - p_a) \log(s + p_{atm})$$

where e is the void ratio, and σ , s , p_a , and p_{atm} are, respectively, the vertical stress, the suction, the air pressure, and the atmospheric pressure, all expressed in MPa.

6.7. Saturated hydraulic conductivity

For the bentonite S-2, an empirical relationship was obtained, from saturated specimens, for the coefficient of permeability as a function of the dry density, expressed by the equations (Section 2.5.8.)

$$\log k = -8.14 \rho_d - 0.72, \\ r^2 = 0.88, 12 \text{ points, for } \rho_d \text{ between } 1.1 \text{ y } 1.4 \text{ g/cm}^3$$

$$\log k = -2.97 \rho_d - 8.29, \\ r^2 = 0.80, 9 \text{ points, for } \rho_d \text{ between } 1.4 \text{ y } 1.9 \text{ g/cm}^3$$

where k is the coefficient of permeability in m/s and ρ_d is the dry density in g/cm³.

For the FEBEX bentonite, CIEMAT has made a set of tests of permeability for nominal dry densities of 1.60 g/cm³ and 1.70 g/cm³ (Section 4.4.2.).

Figure 6.3 shows the straight regression lines for the coefficient of permeability of the S-2 bentonite. When points from FEBEX bentonite are added to the figure it can be observed, that the FEBEX bentonite is more impermeable than the S-2. If the average values of the coefficients of permeability for the two bentonites are compared, the ratio of the values of the S-2 divided by those of the FEBEX is 1.4 to 1.5 for the nominal dry densities of 1.60 g/cm³ and 1.70 g/cm³, respectively. A test made by UPC-DIT for the FEBEX bentonite gave a coefficient of permeability similar to the average value of S-2; nevertheless, it seems that the FEBEX bentonite has a tendency to be more impermeable than the S-2 but the difference is small for such low values of the coefficient of permeability; it is even smaller than the dispersion in a set of tests.

6.8. Thermal conductivity

The regression curves of the values of thermal conductivity as a function of the degree of saturation, for both the bentonite S-2 (Section 2.5.11.) and for FEBEX (Section 4.4.3.) are represented in Figure 6.4. Also represented are all of the values, distinguished by the range of dry densities, of the specimens tested. A good correlation of the sigmoidal type (Boltzmann) was obtained for the two bentonites by means of the equation

$$\lambda = A_2 + \frac{A_1 - A_2}{1 + E^{(S_r - x_0)/d_x}}$$

where λ is the thermal conductivity in W/m · K, S_r is the degree of saturation, A_1 is the value of λ for

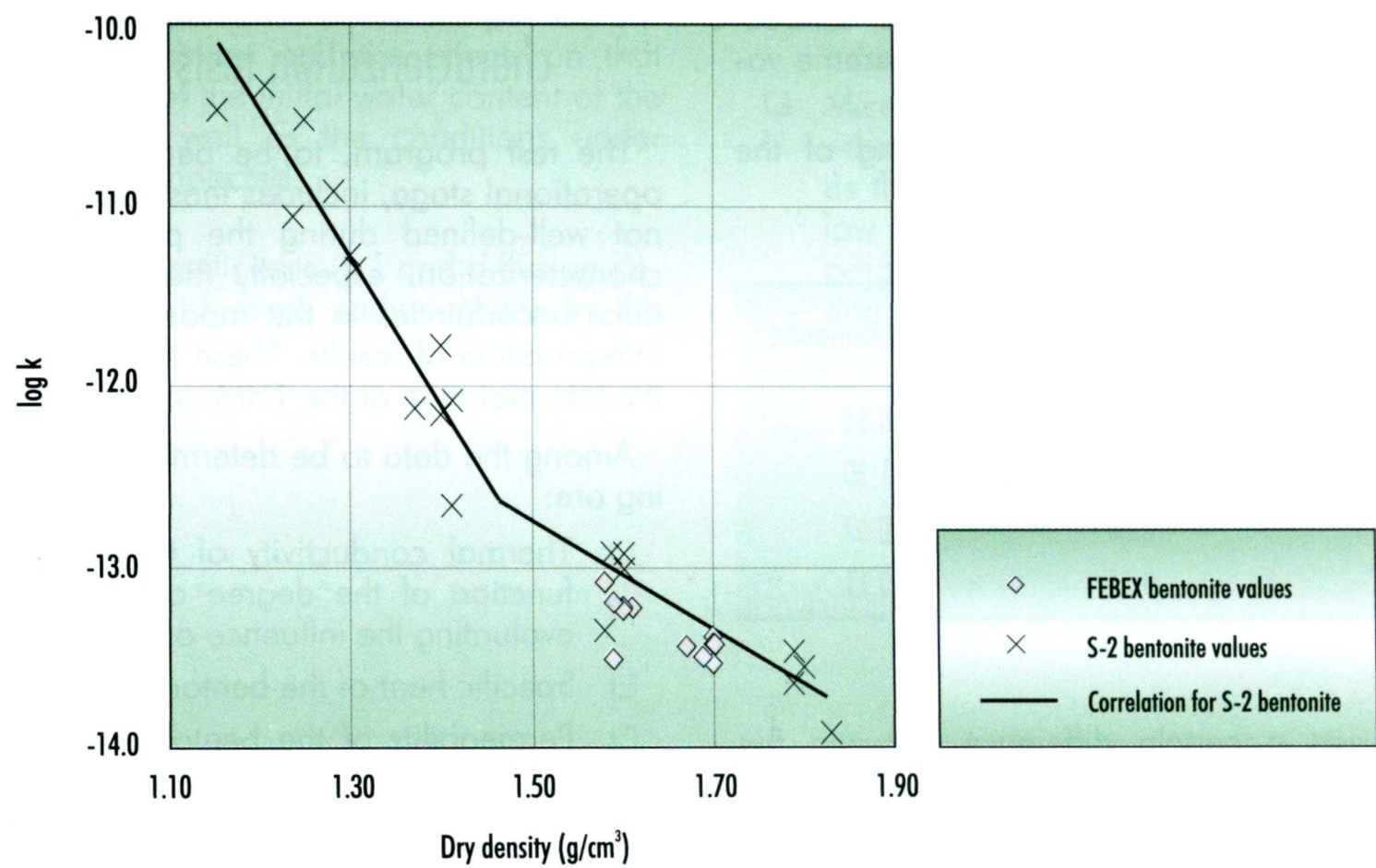


Figure 6.3: Coefficient of hydraulic conductivity vs. dry density.

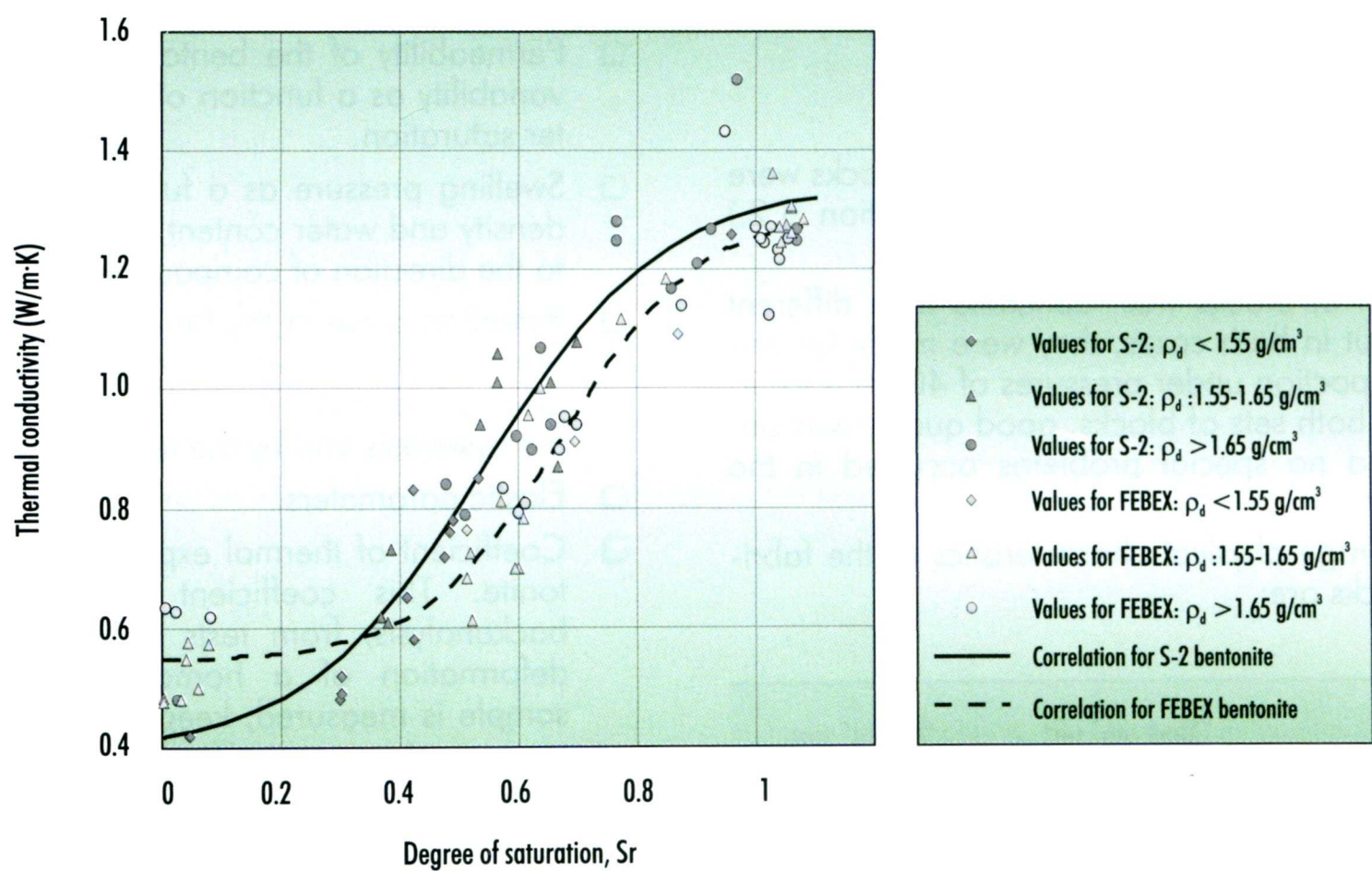


Figure 6.4: Thermal conductivity as a function of degree of saturation.

$S_r = 0$, A_2 is the value of λ for $S_r = 1.0$, x_0 is the degree of saturation for which the thermal conductivity is the average value between the extreme values, and d_x is a parameter.

The parameters obtained in the fitting of the curves are the following:

	Bentonite S-2	FEBEX Bentonite
A_1	0.39	0.55
A_2	1.34	1.30
x_0	0.54	0.67
d_x	0.15	0.11

There exists a certain difference between the curves, more notable in the lower values of the degree of saturation. No clear evaluation of the differences is possible: on one hand, because the thermal conductivity increases not only with the water content but with the dry density; and, on the other, because, for the bentonite S-2, there are only two points for the lower extreme of the degree of saturation. But taking into account all the points, it seems that this parameter differs very little between the two bentonites.

6.9. Bentonite blocks

From the FEBEX bentonite two sets of blocks were fabricated: for the “mock-up” test (Section 5.2.) and for the “in situ” test (Section 5.3.).

Each set of blocks was fabricated in a different factory, but in both cases, they were made by uniaxial compaction under pressures of 40 MPa to 50 MPa. For both sets of blocks, good quality was obtained and no special problems occurred in the fabrication.

The average physical characteristics of the fabricated blocks are:

	“Mock-up” test	“In situ” test
Weight (kg)	25.3 to 25.8	21.3 to 23.1
Dry density (g/cm ³)	1.76 to 1.82	1.69 to 1.70
Water content (%)	13.1 to 14.2	13.1 to 14.9

6.10. Outline of operational stage characterization tests

The test program, to be performed during the operational stage, includes those aspects that were not well-defined during the previous phases of characterization, especially those that can introduce uncertainties in the modeling and/or in the interpretation of results. These tests are needed for the interpretation of the THM and THG tests.

Among the data to be determined for the modeling are:

- ❑ Thermal conductivity of the bentonite as a function of the degree of water saturation, evaluating the influence of dry density.
- ❑ Specific heat of the bentonite.
- ❑ Permeability of the bentonite to water, as a function of the void ratio in a wide range of densities; and the effects of temperature and of anisotropy.
- ❑ Permeability of the bentonite to water as a function of the degree of water saturation, to be determined from backanalysis of specific tests.
- ❑ Water vapor diffusivity in the bentonite, to be determined in an indirect way from the infiltration tests.
- ❑ Permeability of the bentonite to gas and its variability as a function of the degree of water saturation.
- ❑ Swelling pressure as a function of the initial density and water content; and the effect due to the direction of compaction.
- ❑ Retention curve of the bentonite, including its extreme ends (at very low and very high degrees of saturation) and how it is affected by the hysteresis and by the temperature.
- ❑ Elastic parameters.
- ❑ Coefficient of thermal expansion of the bentonite. This coefficient is obtained, by backanalysis, from tests in which the axial deformation of a homogeneously heated sample is measured, keeping the global water content constant.

Other basic characteristics of the bentonite that were still to be determined during the pre-operational stage, the knowledge of which are necessary to understand the behavior and to perform the THG modeling, are the following:

- Chemical composition of the pore water extracted from the bentonite by compaction under high pressure; and the effects on that composition of the initial water content of the bentonite as well as the conditions under which it is compacted.
- Distribution coefficients (K_d) and diffusion coefficients (D) of some radionuclides in the powdered bentonite (batch tests) and in compacted samples (cell tests) to evaluate the adsorption capacity.
- Microstructure and texture of the bentonite—that can determine aspects of its behavior, as the difficulty of dispersion or the relatively low plasticity—will be studied by means of porosity determination as well as scanning and transmission optic microscopy.

7

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