Poster [IGE]

Investigation on Geological Environments
(S, O, SR) ISOTOPIC CONSTRAINTS ON THE DIAGENETIC EVOLUTION OF THE CALLOVO-OXFORDIAN FORMATION AT THE MEUSE/HAUTE-MARNE URL SITE

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The Callovo-Oxfordian clay formation (COX) in the Meuse / Haute-Marne URL, located at Bure in the north-eastern part of the Paris basin was studied for its mineralogy, geochemistry and physical properties (Gaucher et al., 2004). Its conditions of sedimentation and diagenesis were characterized from the study of the clay fraction (Rousset & Clauer, 2003). This work presents new mineralogical and (S, O, Sr) stable isotope data on infilling and concretions preserved in the COX clay formation, in order to bring new constraints on the sedimentary and diagenetic evolution of the COX formation.

Various types of small concretions, generally with pyrite, occur at several horizons. Bioturbations related to annelids are observed and provide evidence of the biological activity during the sedimentation and early diagenesis. They are mainly infilled by frambooidal pyrite with minor organic matter. Rare early concretion enclose uncrushed ammonites are preserved in the claystone of the lower part of the COX formation. Elsewhere concretions correspond to horizons rich in crushed fossils and started to form after some compaction; consequently they occur later in the diagenesis of the argilites. Two types of infilling are distinguished and seem to depend on the lithostratigraphy. Infilling of pyrite + calcite ± celestite are observed in uncrushed ammonites and horizons rich in crushed shells of the lower part of the COX clay formation (C2b1, C2b2). The pyrite is the first phase formed from the fluid, intimately with organic matter. The remained porosity is filled by coarse-grained automorphic calcite and celestite. Infilling of dolomite ± celestite are observed in horizons rich in carbonates and in crushed shells of the upper part of the COX formation (C2d, C3a). Dolomite occurs as coarse automorphic crystals surrounding the border of the cavities, whereas celestite fills the remained porosity, showing the successive crystallization of dolomite and celestite. Most of these dolomite ± celestite infillings are reworked by a later phase of diagenesis marked by the deposition of chaledony and micritic calcite.

The preliminary (S, O, Sr) stable isotope study was performed on pyrite and celestite from different types of infilling and concretion. The $\delta^{34}\text{S}$ values of pyrite range between -32 and +72‰ (vs CDT). They are consistent with a bacterial sulfate-reduction. For a proper interpretation of S isotopes, it is also important to take into account the temperature of formation and the open-closed system behaviour of S species during precipitation. The latter is necessary for evaluating the S isotopic fractionation. The following assumptions were considered for both modelling open and closed system: 1) pyrite only results of bacterial activity; 2) temperatures are around 40°C, biomarkers such as aragonite being preserved; 3) the $\delta^{34}\text{S}$ of dissolved sulfate in porewaters is initially similar to the marine sulfate at the Callovian-oxfordian times; and 4) the $\delta^{34}\text{S}$ of pyrite precipitated by sulfate reduction was not modified by diagenetic processes. Three types of systems: system open to $\text{SO}_4^{2-}$, system closed to $\text{SO}_4^{2-}$ and $\text{H}_2\text{S}$, and system closed to $\text{SO}_4^{2-}$ and open to $\text{H}_2\text{S}$, were modeled following Ohimoto and Rye (1979), and represented in the Figure 1.

Sulfur isotopic composition of pyrite from bioturbations and from early formed concretions are representative of the early diagenesis, i.e before the compaction. Pyrite $\delta^{34}\text{S}$ in most of the C2b1 unit exhibits negative values (-38 to -22‰), suggesting pyrite formed by bacterial reduction of marine sulfates in an open $\text{SO}_4^{2-}$ system. At the limit C2b1/C2b2 and in the C2b2 unit, the $\delta^{34}\text{S}$ values of pyrite shows larger variations from -32 to +34‰, suggesting changes in the conditions of deposition with a system closed to $\text{SO}_4^{2-}$ but still open to $\text{H}_2\text{S}$ aqueous mobility.
Sulfur isotopic composition of pyrite from a concretion corresponding to a horizon rich in crushed fossils and formed after compaction in the lower part of the COX formation provides information on the late diagenetic evolution of the COX clay formation. The extremely high δ34S value of +74 ‰ are only possible in a system closed to SO4\(^{2-}\) while remaining open to H₂S diffusion.

In the both cases, the closure of the system to SO4\(^{2-}\) indicates that the sediments are no more directly in contact with marine water. That could either due to a period of regression, or to the deposition of new sediments which restricts the contact surface between the oldest sediments and marine water. In the case of pyrite created by bioturbation (formed before the compaction of the sediments), the δ34S of pyrite tend to increase twice, from C2b1 to the top of C2b2 and then again above. These variations at the scale of the lithostratigraphic column preferentially reflect the conditions of early diagenesis and suggest a regression period during the C2b2. That is in agreement with previous works (Rouset, 2002). In the case of the pyrite from a concretion corresponding to a horizon rich in crushed fossils, the extremely high δ34S of pyrite reflects the conditions after compaction in the lower part of the COX formation, and suggests that pyrite precipitated by bacterial reduction of highly fractionated residual sulfates dissolved in late trapped porewaters.

Sulfur and oxygen isotopic compositions of late diagenetic celestite range respectively between +22.5 and +31.4 ‰, and +19.5 and +21.2 ‰. δ34S values are higher than that of Upper Jurassic sea water (Claypool et al., 1980). However the 34S and related 18O isotopic enrichment of celestite can be representative of a residual sulfate after bacterial reduction, taking account the previous data on pyrite.

87Sr/86Sr of celestite collected in the lower part of the COX formation ranges between 0.706872 and 0.707040, and can be compared with the signature of the Jurassic seawater (Jones et al., 1994). 87Sr/86Sr of a celestite collected in the upper part of the COX formation (C2d), with a value of 0.707373, is more radiogenic than Jurassic seawater, suggesting an input of radiogenic strontium from Rb-bearing minerals (micaceous clays or feldspars) or from an external source.

References:
SCALE EFFECT AND HETEROGENEITY OF HYDRAULIC CONDUCTIVITY OF SEDIMENTARY ROCKS AT HORONOBE URL SITE

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INTRODUCTION

Representative elementary volume is an important factor on hydraulic investigations regarding the geological disposal of high-level radioactive waste because it is strongly relevant to the scale of the environmental model and groundwater flow simulation for the use of safety assessment. For example, a large-scale groundwater flow simulation can provide the direction and the amount of groundwater flow but it is not suitable to show migration of radionuclides based on “real velocity”, instead of Darcy’s velocity, which represents a smaller-scale behavior. This paper discusses the scale effect, the heterogeneity and porosity-dependence of hydraulic conductivities of sedimentary rocks obtained by the various hydraulic investigations using deep boreholes drilled in the Horonobe Underground Research Project (Goto & Hama, 2003; Matsui et al., 2007) in Japan.

HYDRAULIC INVESTIGATIONS USING DEEP BOREHOLES

The hydraulic investigations using eleven deep boreholes (HDB-1 to 11) include build-up tests, hydraulic packer tests, fluid loggings (flow meter loggings and flowing fluid electric conductivity loggings) and long-term monitoring of hydraulic pressure. In addition, permeability tests using the core samples in laboratory have been performed.

Build-up tests were performed to obtain the rough value of hydraulic conductivities of a half- or a third-length of the boreholes (about 100m to 400m). Hydraulic packer tests were aimed at obtain-ing the hydraulic parameters of both representative rock mass and the flow points detected by the fluid loggings performed prior to the packer tests. The intervals of the packer tests were set up about 10 to 100m. On the other hand, laboratory permeability tests were aimed at getting the hydraulic parameters of intact rocks.

Figure 1 shows the vertical distribution of hydraulic conductivities of the Wakkanai formation that consists of siliceous mudstone, one of the main targets of the study. Hydraulic conductivities obtained by the laboratory tests are lower than those obtained by the in situ tests by more than 1 order of magnitude. And they are almost between $10^{12}$ and $10^{13}$m/s independent of depth. Besides, with regard to the packer tests, the hydraulic conductivities vary widely ranging from $10^{11}$ to $10^{12}$m/s and the domains including the flow points are more permeable than those without flow points by 2 to 4 orders of magnitude in hydraulic conductivity. Build-up tests still have a wide range of hydraulic conductivity but show smaller variance than the packer tests. A larger-scale volume generally shows a smaller variance. But it is difficult in practice to perform a laboratory test with fracture that must be high permeable.

While the Wakkanai formation has high scale effect and heterogeneity in hydraulic conductivities, the contrasts of in situ and laboratory values of the Koetoi and the Yuchi formations are less obvious, where
the Koeto formation is diatomaceous mudstone and the Yuchi formation is fine to medium grained sandstone.

**DISCUSSION**

In order to explain the variance, or heterogeneity, of hydraulic conductivities especially of the Wakkanai formation, the influence of fracture zones on permeability needs to be taken into consideration. Although the authors (Kurikami et al., 2006) mentioned the relationship between the fracture zones and permeability in a previous study, the discussion was not adequate. More detailed investigations on fracture zones and their permeability should be performed.

**REFERENCES:**


**Figure 1:** Hydraulic conductivities of the Wakkanai formation.

**Figure 2:** Hydraulic conductivities vs. porosity (laboratory tests).
INVESTIGATION AND MODELING OF 3D7 DISTRIBUTION OF WATER CHEMISTRIES IN HORONOBE, HOKKAIDO, JAPAN

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INTRODUCTION

The Japan Atomic Energy Agency (JAEA) has undertaken the underground research laboratory (Horonobe URL) project at Horonobe-cho, Hokkaido, Japan since fiscal year 2000. The project has now completed the surface based investigation (for ca. 5 years) consisting of airborne survey, geophysical survey, geological investigation, and borehole drilling investigations. This paper discusses the process to develop conceptual and numerical hydrochemical models based on above field investigations.

BACKGROUND

The Horonobe URL site (≈3 × 3km) is located in the eastern margin of onshore “Tempoku Basin”. The sedimentary sequences consist of the Miocene Masporo and Wakkanai formations, and the Pliocene Koetoi and Yuchi Formations. The fold and fault system in the basin has north-south trend. The two main faults in the area are the Horonobe and Omagari faults. The Wakkanai and Koetoi formations are distributed are mainly composed of diatomaceous mudstone. The mudstone is divided into two depending on degree of the crystallinity of silicate mineral (i.e. opal-A and opal-CT).

The hydraulic tests performed in all of 11 boreholes drilled from 2001 to 2006 used a combination of a pulse, slug, and pumping tests, including groundwater sampling. The test sequences were selected depending on hydraulic conductivity of test interval. Laboratory hydraulic tests using cores have been carried out as well. The hydraulic conductivities are range from 10⁻¹¹ to 10⁻³ m/sec. The results of tests also suggested that hydraulic conductivity in the Wakkanai, Koetoi and Yuchi formations generally decrease with depth, possibly due to closure of conductive fractures.

Hydrochemical analyses have been carried in 10 deep boreholes (470-1,020m in depth) in and around the URL site. The data of the borehole (HDB-2), outside of the URL site was excepted. Groundwater samples were obtained by pumping during the hydraulic test from highly conductive zones (i.e. hydraulic conductivity above 10⁻³ m/s) and also by core squeezing method under hydraulic pressure system. The results suggest that shallower parts of groundwater chemistries are Na-HCO₃ dominated fresh water, while deeper parts are Na-Cl dominated water. Present spatial distributions of groundwater chemistries and evolution process can be conceptually interpreted by long-term infiltration of shallow meteoric fresh water from the surface into deeper saline groundwater.

STATISTICAL ANALYSIS AND NUMERICAL MODELLING

M3 (multivariate, mixing and mass balance analysis, which is a multivariate statistical approach, was applied to identify waters of different origins. The groundwater in and around the URL site can be classified into three types; (1) shallow fresh water (i.e. meteoric water), (2) deep saline water and (3) mixtures between shallow and deep groundwater. The 18O and 2H data also support the mixture of the two end-members water. Deep saline water can be interpreted as being derived from fossil seawater that has
been trapped in the formation during sedimentation. However, the concentration of dissolved ions in deep saline water is significantly lower than that in present seawater, even for a sample of deep saline water that is not affected by meteoric water. This dilution can be explained by crystal water release caused the phase transition of silicate minerals from opal-A to opal-CT.

The 3D groundwater chemistry distribution was depicted by the inverse-distance interpolation method. The result shows that the salinity increases with depth on the west side of the Omagari fault (fig.1). The boundaries between saline water and fresh water were found at deeper depth in the east side than in the west side of the Omagari fault. This observation is hypothetically explained by the fact that the Wakkanai formation is exposed directly on the land surface in the east side, while the Koetooi formation that has lower permeability covers it in the west side. The spatial distribution of thicknesses and hydraulic conductivities of the Koetooi formation probably has been controlling the freshness of saline water during long-term periods. It has been shown that the distribution pattern of chloride concentration is well consistent with a 2D resistivity profile obtained by an electromagnetic tomography.

A numerical simulation coupled with hydrogeology was performed to test the consistency of the conceptual model with the observations of water chemistries. The simulation demonstrated the long-term infiltration of shallow water from land surface into deep saline water that changes the saline/fresh water boundary. As a result, the simulation well explains the present characteristics in spatial distribution of water chemistries around the site (fig.2). For example, the simulations show that freshening of salinity in groundwater proceeds more markedly in the east side of the Omagari Fault rather than the west side, as observed in the field. This result suggests that the hydrogeological model is consistent with observed water chemistries. The discrepancy between observed and simulated distributions also suggests the room for further improvement of the model. Currently, the model still includes the uncertainty, which caused mainly long-term changes of hydraulic parameters and boundary conditions such as land surface up.lift; seawater regression/transgression.

CONCLUDING REMARKS
This paper presented our recent efforts on geochemical investigation and modelling in the Horonobe URL project. The demonstrated methodology would be applicable for another site to evaluate spatial distributions of groundwater chemistries at site scale. In the next step of the investigation, transient geochemical changes during URL construction will be monitored. The model presented here will be further verified and improved to explain such transient monitoring data.
PH4: A 250M DEEP BOREHOLE IN TOUNEMIRE FOR ASSESSING THE CONTRIBUTION OF TRANSPORT PHENOMENA TO ASSUMED OVERPRESSURES IN THE TOARCIAN/DOMERIAN SEMIPERMEABLE

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INTRODUCTION
The safety of a radioactive waste repository in a deep geological formation requires the identification of all processes affecting the undisturbed and disturbed geological barrier. The occurrence of overpressures in semipermeables seems to be the rule in most of the well-compacted and deep clayey formations. These overpressures generally result from a combination of processes among which compaction, diagenesis, hydrocarbon migration, tectonic stress, changes in hydrodynamic conditions, long-term transient osmotic effects and others... This brings us to ask the following question: are we capable of identifying all processes responsible for such overpressures and of estimating their respective contributions? It is especially to try answering this question that a new borehole (PH4) has been drilled in the Tournemire Experimental Station from the tunnel ground between October, 2006 and January, 2007.

EXPERIMENTAL CONCEPT
An overpressure of about 50m was measured at Tournemire in 1994 in a deep borehole (ID180) equipped with a double packer device. This device isolated an 80m height test section crossing a water-bearing fractures in the Toarcian argillite (Matray et al., 2007). Such overpressure would be of the same order of magnitude to that measured in the Callovo-Oxfordian argillite in Bure which was attributed to chemical osmosis “that seems to be capable of explaining the amplitude of the measured overpressure” (ANDRA, 2005). In the case of osmotic transfers, overpressures would result in a water movement from a low concentrated solution (aquifers) towards the semipermeable (the Toarcian/Domerain clayey formation), the osmotic transfer being one of the components of the electrochemical flow couplings. The assessment of such transport phenomena therefore requires the acquisition of (i) transport parameters and water activity profiles between the semipermeable and the surrounding aquifers and (ii) pore-water pressure profile.

(i) Transport parameters and porewater compositions
Water activity can be deduced from the chemical composition of interstitial waters. However, direct water collection was impossible due to the very low water content (3-4wt%) and permeability (10^{-14} - 10^{-15} m/s) of the semipermeable. The methodology finally adopted has consisted in developing a geochemical model at thermodynamic equilibrium that uses rock core analysis. It was decided to fully air core the semipermeable (i.e. 200m of argillite and marls) to obtain a complete profile of the water isotopic composition in order to keep the samples as little disturbed as possible (see Savoye et al., in a companion abstract). Every 10m, specific core samples were preserved in cells with epoxy resin for the acquisition of the mobile anion concentration and of the geochemical porosity by radial diffusion. Other core samples were dedicated to on-site petrophysical measurements at 150°C and some others were preserved in aluminium bags under vacuum for their mineralogy, populations of exchangeable cations, organic matter
and other geomechanical properties. At last, specific core samples were preserved in epoxy resin and in triaxial cells for lab experiments dedicated to the acquisition of osmotic parameters like reflexion coefficients.

(ii) Pressure profile
The verification of the occurrence of an overpressure has needed the installation of a multipacker device right after the drilling and the subsequent diagraphies (diameter, video, BIPS, gamma-ray, neutron porosity, seismic acoustic core borehole...). This has been done in February 2007 by installing a Westbay® device isolating 6 test sections: five in the semipermeable and a sixth one in the lower aquifer. Four of the test zones in the clayey formation were selected in unfractured and short (60cm) areas when the fifth was selected to record pressure in a 60m height zone affected by breakouts. This installation has been followed by the realization of pulse tests after several months acquisition of an historic of pressure. The goals of these pulse tests are the acquisition of hydraulic parameters in the test sections and the acceleration of their static pressure recovery.

MODELLING ASPECTS
The structural parameters i.e. the petrophysical and electro-chemical parameters acquired in the experimental process are used in a modeling approach. The osmotic permeability can be determined using various theoretical expressions (Bresler, 1973; Leroy and Revil, 2004). Usually, these expressions require the resolution of an electrical model that reproduces the interactions between the pore solution and the negatively charged surface of clay minerals. The electrical model that is implemented here is a Triple Layer Model (TLM) that is thought to capture with a better accuracy than the double diffuse layer models these electrical interactions. We developed a 2D-model of coupled flow using the classical formulation for these transfers (see e.g. Gonçalvès et al. 2004; Revil and Leroy, 2004; Gueutin et al., 2006). This coupled flow model includes a TLM module that provides values for the so-called osmotic efficiency. This code is used at the sample scale as well as at the formation scales for the interpretation of the experiments.

Other transient processes than osmosis can be evaluated as already attempted by Gonçalvès et al. (2004). Some variations in the hydraulic boundary conditions can be tested through a classical hydrogeological model. The geological time scale variation in tectonic stress can also be tested introducing a source term in the equation of diffusivity as shown by e.g. Gonçalvès et al. (2004). Provided that a temporal evolution of this tectonic stress can be traced, this simulation can be processed using a classical fluid flow model.

References:


LATERAL VARIABILITY OF MINERALOGY AND PORE WATER CHEMISTRY OF THE BOOM CLAY

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INTRODUCTION

In Belgium, the Boom Clay in the Mol-Dessel region is studied as a reference host formation, respectively reference site, for the geological disposal of high-level and long-lived radioactive waste. The Boom Clay has been studied for many years and its lateral continuity is well-known. However, most of its properties have been investigated at the Mol-Dessel reference site and needed to be further investigated at other locations to allow extrapolation over a larger zone. The Essen-1 borehole, drilled in 2006, and situated at about 50 km to the northwest of Mol, provided the opportunity to analyse geomechanical, chemical and migration properties of the Boom Clay. This paper describes the geochemical properties of the Boom Clay in the Essen-1 borehole, i.e. the mineralogy and the pore water composition, and compares it with the Mol-Dessel reference site.

SAMPLING AND ANALYSES

Ten samples were selected in the Boom Clay of the Essen-1 borehole for analyses of the mineralogy and the pore water composition. The sampling and preservation of the clay cores was performed in such a way that all possible geochemical perturbations were minimised.

Pore water sampling was done by mechanical squeezing of clay core samples. Sample preparation was performed in a nitrogen-filled glove-box (oxygen level < 10 ppm). All samples were squeezed at a constant pressure of 30 MPa during one week. The chemical composition and the stable isotope composition of the pore water was then determined and compared to the reference composition at the Mol-Dessel site.

The whole-rock and clay fraction material were used to investigate the mineralogy of the sediment in the Essen-1 borehole. The samples were grind in RETSCH ball agate mill and sieved to $\leq 0.125$ mm. The clay fraction was separated from the rock matrix by removing carbonates, organic matter and Fe, Mn-oxihydroxides followed by sedimentation and dialysis. The powders and oriented specimens in air-dried and ethylene-glycolated state were measured using an XRD Phillips X’Pert system.

BOOM CLAY PORE WATER COMPOSITION IN THE ESSEN-1 BOREHOLE AND COMPARISON WITH THE MOL-DEssel REFERENCE SITE

The Boom Clay pore water composition in Essen is clearly different from the reference Boom Clay pore water composition at the Mol-Dessel site (De Craen \textit{et al.}, 2004; De Craen \textit{et al.}, 2006). The most important differences are the type of pore water, the salinity, and the vertical variations in chemical composition. At the reference site, the Boom Clay pore water is a NaHCO\textsubscript{3} – type water, while in Essen, it is a NaCl – type water. At the reference site, the salinity of the Boom Clay pore water is rather low (salt content $< 1300$ mg/l). In Essen, the salinity is ranging from 1470 mg/l at the top to 7249 mg/l at the bottom with an average salt content of 4093 mg/l water. The trend of continuous enrichment in salinity from the top to the bottom of the clay is present for most anions (Cl\textsuperscript{-}, SO\textsubscript{4}\textsuperscript{2-}, Br, HCO\textsubscript{3}-, I) and for Na. The salinity increase corresponds to a progressive change from a 6 mM Na(HCO\textsubscript{3}-Cl) – type water at the top to a 108 mM NaCl – type water at the bottom. An important component of these salts is Cl\textsuperscript{-}. While only 26 mg/l Cl\textsuperscript{-} is present in the reference Boom Clay pore water at the Mol-Dessel site, up to 3100 mg/l Cl\textsuperscript{-} is measured in the pore water in Essen. Another major component of the Boom Clay pore water in Essen is
SO$_4^{2-}$. In the reference Boom Clay pore water at the Mol-Dessel site, the SO$_4^{2-}$ content is lower than 10 mg/l. In Essen, up to 790 mg/l SO$_4^{2-}$ can be present. The trend of increasing salinity with depth, which is recognized in the Boom Clay in the Essen-1 borehole, is further continued in the underlying aquifers. The underlying aquifers are also NaCl – type waters with increasing salinity with depth. The aquifer above the Boom Clay (i.e. the Berchem Sands) is a (Na-Ca)(HCO$_3$-Cl) – type water and is clearly different from all other samples in the Essen-1 borehole. In Mol, such a trend is not present. In general, the Boom Clay pore water composition is rather homogeneous at the Mol-Dessel site. Some very small variations (still the same type of water) are sometimes recognised, but these are not related to depth.

The stable isotope composition of the Boom Clay pore water in Essen is characterised by $\delta^2$H values ranging from -47.5 ‰ vs VSMOW at the top to -39.3 ‰ vs VSMOW at the bottom of the Boom Clay. $\delta^{18}$O values range from -6.41 ‰ vs VSMOW at the top to -5.74 ‰ vs VSMOW at the bottom of the Boom Clay. Thus, with increasing salinity towards the bottom of the clay, both $^2$H and $^{18}$O are enriched in the pore water. These data suggest that these pore waters are mainly from meteoric origin, but some mixing with water of marine origin is visible. The mixing with water of marine origin explains the higher salinity and the enrichment in $^2$H and $^{18}$O in the Essen pore waters compared to the pore waters in Mol.

BOOM CLAY MINERALOGY IN THE ESSEN-1 BOREHOLE AND COMPARISON WITH THE MOL-DEssel REFERENCE SITE

The Boom Clay in Mol consists of clay minerals, quartz, K-feldspar, Na-plagioclase, pyrite and carbonates. The clay mineralogy is dominated by kaolinite, illite, mixed-layered illite-smectite and chlorite. In the vertical profile of the Boom Clay at the Mol-Dessel site, the mineralogy is quite homogeneous, differing only in relative abundances of the individual mineral phases. The quantitative variations in the mineralogical composition fairly reflect the alternations between clayey silt and silty clay layers. The total non-clay content was found to vary in the range of 41-77 weight %, the total clay content from 23-59 weight %.

Preliminary results on the Essen-1 borehole show that the overall mineralogical composition of the Boom Clay is very similar to the reference site, but a detailed clay mineralogical study is still in progress. Differences might be expected in the mineralogy of the clay constituents, since earlier studies have shown that some minor lateral variations in the clay mineralogical composition exist (Laenen, 1997). These lateral variations relate to different source areas of the sediment at different locations in the depositional environment.

CONCLUSIONS

Most of the Boom Clay properties have been studied at the reference site in the Mol-Dessel region. In order to extrapolate these properties over a larger zone, the Boom Clay were studied in the Essen-1 borehole. In general, the mineralogical composition of the Boom Clay is comparable between the two sites. Preliminary results suggest some small variations in clay mineralogy, which relate to different source areas of the sediment at different locations in the depositional environment. The Boom Clay pore water composition in Essen is clearly different from the reference Boom Clay pore water composition at the Mol-Dessel site. The most important differences are the type of pore water, the salinity, and the vertical variations in chemical composition. The higher salinity and the increasing salinity with depth are explained by the mixing with water of marine origin.

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References


**36Cl IN GROUNDWATERS OF OXFORDIAN AND DOGGER LIMESTONES OF THE EASTERN PARIS BASIN: IMPLICATIONS FOR OLD GROUNDWATER DATING**

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**INTRODUCTION**

In the eventuality of containment barrier failure in deep underground nuclear waste storage scheme, the estimation of groundwater residence time is essential to evaluate the time scale of radionuclide dispersion by advection. Diffusive vertical transfer of anions out of the clay layer in the Oxfordian limestones of the Paris Basin was shown (Lavastre et al, 2005) and the superimposed advective transfer in limestones surrounding the clay layer would contribute to a potential dispersion of the radionuclides as anions. Beyond 30 ka, the limit of 14C dating, groundwater residence time is rather difficult to estimate. With a half-life of 301 ka, 36Cl is particularly appealing for dating groundwaters on the time scale of 10⁵ to 10⁶ years. 36Cl is produced in the atmosphere by spallation of argon nuclei by cosmic rays. After entering the hydrologic cycle by dissolution in precipitation and infiltration, groundwater residence time can be estimated on the basis of cosmogenic 36Cl radioactive decay. Chloride being a conservative ion in solution, changes in 36Cl signal due to water rock interaction is limited and decreasing 36Cl concentration may be attributed to radioactive decay (Phillips 2000). However the cosmogenic origin of groundwater 36Cl has to be ascertained, the input ratio quantified, and potential contribution of deep chlorine-36 needs to be accounted for. This study investigates old groundwaters from the confined Oxfordian and Dogger limestone formations surrounding Callovo-Oxfordian clay layer in the vicinity of the Andra Underground Research Laboratory (URL), east of the Paris Basin.

**RESULTS AND INTERPRETATION**

Dogger and Oxfordian formation waters were collected during the Andra 2003-2004 sampling campaign. Groundwaters of 7 Porous Horizons (PH) in the Oxfordian limestones were sampled during the excavation of the URL main shaft. Another 15 groundwaters were sampled in additional 8 boreholes in the Oxfordian and Dogger formations in the vicinity of the URL, either by slow pumping rate at the depth of porous horizons during borehole excavation (‘Diapo’), or as long term pumping (LTP) after completion of the borehole. The AgCl precipitate was prepared at CEREGE and 36Cl/Cl ratio was determined by accelerator mass spectrometry at the Lawrence Livermore National Laboratory CAMS while stable chloride concentrations were determined by ion chromatograph at CEREGE. AMS blanks were approximately one order of magnitude lower than the samples and precision on stable chlorine concentrations reach 3% precision.

Dogger groundwater 36Cl/Cl ratio (1 to 5 x 10⁻¹⁵ at.at⁻¹), similar to that at secular equilibrium in this formation (2.3 to 6.5 x 10⁻¹⁵ at.at⁻¹), can be explained either by i) mixing with “old” chlorine in equilibrium with the matrix or by ii) chlorine-36 decay. The later hypothesis, while unlikely, would suggest groundwater with age over 1.2 to 1.5 Ma (i.e. 4 to 5 times the 36Cl half-live).

Most Oxfordian groundwaters seem to follow a mixing trend on a 36Cl/Cl ratio vs chlorine plot, between a low Cl and high 36Cl/Cl end-member and a high chloride end-member with low 36Cl/Cl ratio tending toward the 36Cl/Cl ratio at secular equilibrium (6 to 10 x 10⁻¹⁵ at.at⁻¹) (Fig. 1). Mixing could occur i) with
chlorine from either Dogger or even Triassic groundwaters by advection through fractures, ii) either with chlorine diffusing from the underlying Callovo-Oxfordian clay layer separating both limestone formations, and/or iii) between groundwaters from several porous horizons (PH) in the Oxfordian limestone presenting varying $^{37}$Cl signature. Chlorine-37, which fractionates during chloride diffusion, provides further information on the mixing process. Oxfordian groundwater $^{37}$Cl ranging between -2.13 and -0.07 $\%_{\text{e}}$ vs SMOC supports the hypothesis of chloride diffusion from the underlying Callovo-Oxfordian clay layer (Lavastre et al, 2005). However on a $^{37}$Cl vs $^{36}$Cl/Cl ratio graph (Fig 1b), two of the high $^{36}$Cl/Cl ratio and low chloride concentration groundwaters, EST 351 and EST 321, show little $^{37}$Cl fractionation, with $^{37}$Cl similar to that of the shallow Tithonian groundwaters (-0.7 to +0.3 $\%_{\text{e}}$ vs SMOC), suggesting that they are not or little affected by chlorine diffusion and might still be considered for dating.

In the hypothesis of mixing with deep $^{36}$Cl, no groundwater ages can be calculated. In the hypothesis that EST321 is not affected by mixing and if we can assume that EST321 is representative of the input signal, straight radioactive decay between EST321 and EST351 can be considered. Taking into account the $^{37}$Cl in situ production, ages in the order of 400±70 ka and 450±70 ka can be calculated for EST351 393m and “LTP” respectively. However EST321 does not necessarily represent the input signal. Considering a theoretical $^{36}$Cl input signal in the precipitations, based on calculated $^{36}$Cl fallout (Phillips 2000), leads to ages ranging between 200±80 to 570±160 ka depending on the considered initial conditions including the rate of evapotranspiration and potential variations of the $^{36}$Cl/Cl cosmogenic production. All these emphasize that it is essential to i) better define the input signal to the system through the investigation of groundwater chlorine-36 signal toward the recharge area and ii) identify additional groundwater sample not or little affected by mixing. Measurement of chlorine-36 concentration in modern precipitations in the studied area would also contribute to assist in better defining the input signal.

References:
STATISTICAL PREPROCESSING AND ANALYSES OF HYDROGEO-METEOROLOGIC TIME SERIES
IN THE PP EXPERIMENT OF MONT TERRI (METHODOLOGY AND FIRST RESULTS)

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HYDROGEOLOGIC AND MEASUREMENTS CONTEXT
The signal processing methods illustrated here were applied to measurements obtained over a period of ten years in the Opalinus clay at the URL Mont Terri in Switzerland. Absolute pore pressures are monitored in isolated chambers of the BPP1 borehole (niche PP). This borehole was selected because its longest times series of pore pressure over a period of about ten years (1996 to 2005). These time series are compared to those of atmospheric pressure, temperature, and relative humidity measured in the exploration gallery.

OBJECTIVES
The objective of this work is to exploit the set of time series at the PP borehole in order to:

- Evaluate direct and coupled transfer processes involving fluctuations of pore pressure under the influence of “natural forcing” (earth tides, barometric fluctuations, rainfall, humidity...);
- Estimate the hydraulic properties (specific storativity, porosity...) around the borehole, as well as their evolution in time, and compare them to the hydraulic properties estimated from direct short time hydraulic tests (pulse tests, slug tests).

PRE-PROCESSING OF COMPLEX MULTIVARIATE TIME SERIES
The M' Terri time series are affected by some common defects which need to be detected and corrected. In the case at hand, the problems are as follows: (1) Missing data : there are data gaps of various lengths (e.g., isolated gaps, but also, much longer gaps involving one hundred or more time steps); (2) Variable time step \( \Delta t(i) \) (e.g. : 1 min, 30 min, 4 days...); (3) Outliers and spurious data (aberrations), e.g.: data affected by large measurement errors, measurement bias, or uncontrolled human intervention (for example, negative values of \( p_{\text{ATM}} \)).

A first batch of analyses is conducted on the raw time series in order to detect missing data, variable time steps, and potentially abnormal data. Depending on the magnitude of these problems, various techniques are then applied in order to partially reconstitute and/or homogenize the signals. Among other methods, we used: moving averages and other filters, linear interpolation techniques, autoregressive models, and posterior assessments based on moments and covariance structures. Some of these techniques are empirical; others rely on the theory of stationary random fluctuations. One aspect of the problem is deciding (with reproducible criteria) whether or not to split time series containing long subsets of missing or anomalous data. Splitting algorithms were designed for bivariate time series, in order to allow for cross-covariance and cross-spectral analyses. Such operations are easier if time step is constant, but this is not always the case. Finally, if time step is moderately variable, a “homogenization” of time steps \( \Delta t(i) \) is performed by linear interpolation and extrapolation techniques in order to obtain a new series with constant time step (example given on fig.1).
It is clear that, without pre-processing, only short time series would be available for analysis. Specifically here, the time series available for bivariate statistical analyses would not be much longer than one month, out of a sampling period of about ten years. Without attempts at reconditioning, homogenizing, or reconstituting some of the data, precious information on the behaviour of hydrogeological signals at various time scales would be lost.

STATISTICAL ANALYSES AND INTERPRETATION OF PRESSURE SIGNALS (RECONSTITUTED OR NOT)

After the previous pre-processing, we present various methods for analyzing the statistical properties of the time series in order to characterize the hydraulic behaviour of the porous formation.

- Correlation analyses in the time domain (auto- and cross-correlations, time transfer functions)
- Fourier spectral analyses in frequency domain (simple spectrum, cross-spectrum, spectral gain)
- Multi resolution wavelet analyses.

Figure 1: Example of pre-processing of a 3 day section of $p_{370}(t)$ time series at Mont Terri (27/06/2005-30/06/2005) : comparison of the raw signal with variable $\Delta t(i)$, to the “homogenized” signal with constant $\Delta t_0$ (the chosen time step is $\Delta t_0=3600\ s$).

Figure 2a: Normalized spectrum of $PP_1(t)$ - first difference of relative pore pressure (kPa). Mont Terri 02/08/2002-04/09/2002, $\Delta t = 30\ min$, sampling step $k=1$.

Figure 2b: Multiresolution wavelet analysis: dyadic time scale component 8h (near 12h) – relative pore pressure $PP_1(t)$. Mont Terri 02/08/02-22/08/02, $\Delta t = 30\ min$, $k=1$.

Figures 2(a,b) illustrate some of these techniques using only short sections of the original signals with constant time steps. These analyses enable us to detect and characterize the diurnal relation between piezometry and atmospheric pressure (whence an equivalent “barometric efficiency”) and the semi-diurnal effect of earth tides (whence an approach to specific storativity of clay). A more “mechanistic” hydro-mechanical modelling of these phenomena, coupling pore pressure with external forcing, is also being considered (for more details, see Mallet et al. 2007 in this conference).

In future, we will proceed to more systematic statistical comparisons of processed vs. unprocessed signals in order to assess the added value of signal pre-processing, with a view to characterizing the hydraulic behaviour of clay at various time scales.
HYDRAULIC CONDUCTIVITY OF THE BOOM CLAY IN NORTH-EAST BELGIUM BASED ON FOUR CORE-DRILLED BOREHOLES

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INTRODUCTION
In Belgium, geological disposal of high-level radioactive waste in the Boom Clay Formation is considered as a potential solution, given its various favourable properties as a.o. a very low hydraulic conductivity (about 10^{-12} m/s), strong sorption capacity for radionuclides and self-sealing properties. This Tertiary argillaceous formation is extending in north-east Belgium. It crops out in a small west-east band to the south of Antwerpen and Hasselt and it is slightly dipping into the north-north-eastern direction. At the Mol site, the Boom Clay has a thickness of a little more than 100 m, whereas its top is located at about 186 m depth.

Results on the hydraulic conductivity of this formation are already systematically collected for many years in order to evaluate the transport processes within the host-formation (which is dominated by diffusion) and to simulate the flow and transport within the surrounding aquifer system. These data are therefore processed to provide regional distribution of the hydraulic conductivity. This paper presents the results of numerous hydraulic conductivity values (K) determined in the laboratory, on clay cores from four distant boreholes i.e. Doel-2b, Zoersel, Mol-1 and Weelde-1. These boreholes have increasing depths and thicknesses and are spread over the north-north-east of Belgium in an area of about 1100 km^2. The results are interpreted according to depth and possible hydrostratigraphic subdivisions. Distinction is made between the vertical and the horizontal hydraulic conductivity (K_v and K_h), which allows a determination of the anisotropy at the sample scale. Complementary analyses, as grain-size, are used to extrapolate the vertical profile of hydraulic conductivity and to increase our confidence in the global value of the parameter at the scale of the formation thickness. The global values of K_v and K_h are calculated using harmonic means of the lower scale values of K_v and K_h.

MATERIALS AND METHODS
High quality cores were obtained from the four boreholes, which were cored with a pushing corer device particularly adapted for unconsolidated sediments. Detailed macroscopic description was done on thin splits of the cores and compared with open-hole geophysical measurements. Samples of about 50 cm^2, were taken from well-conditioned cores for the determination of the hydraulic conductivity. The hydraulic conductivity values were obtained, based on Darcy's law, from transport experiments with pulse injection of iodide and tritiated water in confined percolation cells (Aertsens et al., 2004) or by classical permeameters, in the laboratory. Grain-size analyses were performed on the hydraulic conductivity samples or on small samples taken from the thin core splits. The grain-sizes were determined with a sedigraph, after extraction of organic matter and calcium carbonate.

RESULTS AND INTERPRETATION
The four investigated boreholes (Doel-2b, Zoersel, Mol-1 and Weelde-1) present very low hydraulic conductivity values with geometric averages of K_v of about respectively 1×10^{-11} m/s, 9×10^{-12} m/s, 3×10^{-12} m/s and 8×10^{-12} m/s (Aertsens et al., 2003, 2004; Wemaere et al., 2002, 2004, 2005). The K_h value at the sample scale is about twice the value of the vertical one. The hydraulic conductivity can be considered as homogeneous over the vertical section of the Boom Clay; however, looking into details, one can recognise some differences according to typical sub-units of the formation. The Boom Clay has a main
Table 1: Hydraulic conductivity values of the Boom Clay measured on cores from four boreholes in north and north-east Belgium (b: thickness of the unit/sub-unit).

<table>
<thead>
<tr>
<th>Unit</th>
<th>Doel</th>
<th>Zoersel</th>
<th>Mol-1</th>
<th>Weelde</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td>b (m)</td>
<td>$K_v$ (m/s)</td>
<td>$K_H$ (m/s)</td>
<td>b (m)</td>
</tr>
<tr>
<td>Transition zone</td>
<td>–</td>
<td>–</td>
<td>–</td>
<td>22</td>
</tr>
<tr>
<td>Putte + Terhagen</td>
<td>59</td>
<td>7 $10^{-12}$</td>
<td>1 $10^{-11}$</td>
<td>67</td>
</tr>
<tr>
<td>Belsele-Waas</td>
<td>9</td>
<td>2 $10^{-10}$</td>
<td>4 $10^{-10}$</td>
<td>9</td>
</tr>
<tr>
<td>Boom Unit</td>
<td>67</td>
<td>8 $10^{-12}$</td>
<td>6 $10^{-11}$</td>
<td>97</td>
</tr>
</tbody>
</table>

part, about 60 to 70 m thick, in the middle of the formation, consisting of the members of Putte and Terhagen, with the lowest average value of $K$ and the lowest variations according to depth (i.e. less than one order of magnitude). It is overlain by the Transition-zone unit, which presents a slightly higher average value of $K$, in Zoersel and Mol but also slightly higher variations with depth. This unit is absent in Doel where it has been eroded but it is nearly twice as thick in Weelde (40 m) with a more significant increase of the $K$ average. Finally, the 9 to 17 m thick Belsele-Waas Member, at the bottom of the formation, has both a higher average value of $K$ and the highest variation with depth (i.e. one or two orders of magnitude). The $K_v$ and $K_H$ values of the sub-units allow to propose an equivalent $K_v$ and $K_H$ at the scale of the formation thickness, based on harmonic means (see Table 1).

The differences between the sub-unit values are mainly related to differences in grain-sizes. The Putte and Terhagen Members are clayey silt and silty clays with a very low sand content, while the Belsele-Waas Member can clearly be seen as a clayey silt with more than 5% sand. Grain-size analyses were used as soft data to provide much more detailed profiles of hydraulic conductivities and increase confidence in the proposed $K$ ranges. These results are further integrated together with results from the Mol-site and other locations, (Wemaere and Marivoet, 1997), to propose a realistic and detailed distribution of the hydraulic conductivity of the Boom Clay over its depositional area, in Belgium.

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References:


A NEW APPROACH OF LABORATORY PERMEABILITY MEASUREMENT OF VERY TIGHT ROCKS: EXPERIMENTAL DATA OF BCF, MECSEK MTS, SW HUNGARY

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INTRODUCTION
Research aimed at the geological disposal of HLW produced in the Paks Nuclear Power Plant (Paks NPP) has focused on the Upper Permian Boda Claystone Formation (BCF) since 1993. The horizontal and vertical dimensions of the BCF, one of the main part of the terrestrial – fluvial – lagoonal Permian sedimentary sequence of the W Mecsek Mts., are approx. 150 km² and 700-900 m respectively. Especially, reddish brown albitic claystone alternating with siltstone, sandstone, dolomite and albite could be distinguished. Shallow-water lacustrine environment under semi-arid to arid climatic conditions with dominantly oxidizing depositional and early diagenetic environments are presumed. The peculiar physical and chemical character of the investigated rocks induced applying many new methods and experimental development, because the traditional methods could not be used. One of the new methods used was the pressure pulse decay (PPD) technique to estimate permeability.

METHODS AND EXPERIMENTS
In the early 1990s an exploration shaft, the so-called Alfa Shaft was excavated from the former uranium mine 1000 m underground. This was one of the first and the deepest URLs of the world to investigate argillaceous rocks. During the operation of the URL, some laboratory measurements were executed to determine permeability under stress and at a given temperature. However, the unreliability and the non-robust and time-consuming character of the measurements made it necessary to find new ways.

A new experimental PPD permeameter was built to measure both gas and water permeability. The principle of the measurement is that the time of pressure pulse decay between the two sides of a core depends on the permeability of the rock. Theoretical background is given by many articles, e.g. Jones (1997). The aim was to investigate cores in quasi reservoir conditions (35 °C, 6-7 MPa, N₂ gas/distilled water) in a relatively short time where the uncertainty of the results can be estimated.

Four core samples from the underground boreholes of the Alfa shaft (BAF-1 to 4) were investigated. These cores represent the dominant rock type of BCF, the albitic claystone composed of illite and/or muscovite (30-40 %), chlorite (5-10 %), authigenic albite (25-40 %), quartz (5-10 %), carbonates (5-15 %, generally calcite or dolomite), hematite (5-10 %) and inconsiderable amounts of smectite, kaolinite and vermiculite. The sample BAF-1 was used to determine the physical behaviour of rock during cutting. The cores BAF-2 to 4 and the small cores cut from them served for measuring the gas and water permeability and also the pore throat size distribution by mercury porosimetry. Large samples were dried at 105 °C to reach the dry weight (20-30 days) and effective porosity was measured with a He-pycnometer and also estimated by calculation. After that, seven small samples were cut from the large samples, a cubic one for mercury porosimetry and six cylindrical bodies for permeability measurement. These small samples were also dried and porosities were estimated.
RESULTS AND INTERPRETATION

Table 1: A sample of results of porosity, gas and water permeability measurements on BCF cores.

<table>
<thead>
<tr>
<th>ID</th>
<th>diameter [mm]</th>
<th>length [mm]</th>
<th>volume [cm³]</th>
<th>porosity [%]</th>
<th>T [°C]</th>
<th>$p_i$ [bar]</th>
<th>$k_{v,rel}$ [m²]</th>
<th>s [±/± m²]</th>
</tr>
</thead>
<tbody>
<tr>
<td>BAF-2/1</td>
<td>36.290 ± 0.010</td>
<td>9.930 ± 0.000</td>
<td>10.271 ± 0.006</td>
<td>2.12</td>
<td>35.780 ± 0.053</td>
<td>78.895</td>
<td>6.66·10⁻¹⁹</td>
<td>1.6·10⁻²¹</td>
</tr>
<tr>
<td>BAF-2/2</td>
<td>36.284 ± 0.009</td>
<td>10.026 ± 0.005</td>
<td>10.367 ± 0.011</td>
<td>2.12</td>
<td>34.470 ± 0.069</td>
<td>79.948</td>
<td>1.33·10⁻¹⁹</td>
<td>3.1·10⁻²¹</td>
</tr>
<tr>
<td>BAF-3/2</td>
<td>36.310 ± 0.007</td>
<td>10.133 ± 0.032</td>
<td>10.492 ± 0.037</td>
<td>1.16</td>
<td>35.476 ± 0.043</td>
<td>79.255</td>
<td>1.51·10⁻¹⁸</td>
<td>3.5·10⁻²⁰</td>
</tr>
<tr>
<td>BAF-3/3</td>
<td>36.316 ± 0.005</td>
<td>9.929 ± 0.078</td>
<td>10.285 ± 0.084</td>
<td>1.16</td>
<td>35.201 ± 0.042</td>
<td>78.920</td>
<td>7.93·10⁻¹⁹</td>
<td>2.7·10⁻²⁰</td>
</tr>
<tr>
<td>BAF-4/2</td>
<td>35.958 ± 0.015</td>
<td>10.381 ± 0.031</td>
<td>10.542 ± 0.040</td>
<td>2.36</td>
<td>35.199 ± 0.019</td>
<td>78.605</td>
<td>1.02·10⁻¹⁸</td>
<td>3.1·10⁻²¹</td>
</tr>
<tr>
<td>BAF-4/3</td>
<td>35.944 ± 0.017</td>
<td>9.974 ± 0.015</td>
<td>10.020 ± 0.025</td>
<td>2.36</td>
<td>35.312 ± 0.035</td>
<td>79.325</td>
<td>1.49·10⁻¹⁸</td>
<td>6.3·10⁻²⁰</td>
</tr>
</tbody>
</table>

* $p_i$ internal pressure

Finally, pore throat size distribution, gas and water permeability were determined. All cores and experiment results were digitally documented.

RESULTS AND INTERPRETATION

The calculation of matrix porosity – using the weight differences of wet and dry state of the core and the core size regarding the results of He-porosity measurements – gave the only reliable information. The porosity is 1-3 %, which is marginally higher than it was estimated previously. The time-consumption of drying and the fact that the dry core absorbed humidity of the atmosphere very fast drew attention to the importance of the necessity of careful preparation and handling.

The measurements of mercury porosimetry have to be repeated using high pressure mercury porosimeter because more than three-quarter of pores form part of lower mezo- and micropores.

Permeability measurements under reservoir condition show that the constant pressure difference (20-25 bar) between the outside and inside of rubber of Hassler-type core-holder and the constant temperature are very important. A sample of the results of measurements is shown in table 1.

On the basis of all results, the inhomogeneity of permeability can be recognized. On the other hand, tendentious gas permeability decrease was observed at constant temperature when the pressure decreased. The latter has to be studied in more detail because not all reasons of the decrease are clearly defined. Water permeability values are approx. one or two much smaller than gas permeability. During the water permeability measurements the initial weight of fully saturated cores increased. The reason of this phenomenon may be explained by the mineralogical composition.

References: