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# **CEBAMA**

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### **D1.01 Detailed WP1 description of scientific work: compilation of partner descriptions**

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<b>RE</b>	Restricted to a group specified by the partners of the CEBAMA project	
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**ABSTRACT:**

This report gives details of the planned work for each of the participants of work package 1, related to Experiments on Interface Processes and the Impact on Physical Properties. The written descriptions reported here have been provided by the project partners during the project preparation phase during 2014. The information was reviewed and compiled by the WP1 leaders, as authors of this deliverable. The complimentary overview slides of WP1 work were presented by the partners during the Project's Kick-off meeting held in Brussels on 2nd July 2015.

**Keywords:** experimental studies, methods, materials, work description

**RESPONSIBLE:**

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## **1 PARTNER WORK DESCRIPTIONS FROM PROJECT PLANNING**

All participants in WP1 gave an overview of the expected work scope. This was summarized in written format during the project planning phase, with an emphasis on experimental work to be performed and cooperative nature between the partners, materials and methodologies. These summaries are presented in Appendix 1.

## **2 PARTNER WORK DESCRIPTIONS FROM KICK-OFF MEETING PRESENTATIONS**

All participants in WP1 gave an overview of the expected work scope, summarized as slides during the Project Kick-off meeting held in Brussels on 2<sup>nd</sup> July 2015. These summaries are presented in Appendix 2. Note that slides 1-12 present the WP1 overview by the WP leaders, while slides 14-54 are the individual partners' work contributions. It has been noted in each set of the partner's slides that there are no deviations from the scientific and technical scope of the work plan compared to the original Project Plan at the time of proposal submission.

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## APPENDIX 1: Partner Written Summaries

### Appendix to Cebama proposal

#### 1 /KIT

WP 1			KIT
<b>Overall Objectives:</b> KIT performs experiments on interface reactions of low pH cementitious materials in contact with clay-water-systems and quantifies the precipitation/dissolution processes in pores, changes in pore size / distributions and changes in permeability by diffusion experiments.			
<b>Objectives for different tasks (specific to your organization):</b> <ul style="list-style-type: none"> <li>• <b><u>Task 1.1: Establishing known State-of-the-Art, defining boundary conditions for experimental studies and details of analytical methods for sample characterization</u></b>  Preparation of low pH concrete materials, complete hydration (pre-Cebama period), interaction with bentonite water, batch experiments for identification of changes in mineral phases, set-up of diffusion experiments, characterization of the concretes (porosity/mineralogy)</li> <li>• <b><u>Task 1.2: Characterization &amp; experimental studies.</u></b>  Non-equilibrium diffusion experiments with low-pH cement formulations: <ul style="list-style-type: none"> <li>○ Determination of initial diffusion properties with inert tracers (HTO). Derivation of diffusivity properties as function of time.</li> <li>○ Determination of diffusion coefficients and retention properties of relevant radionuclides, e.g. Be, I, Cl and on basis of consultation with other partners (Amphos, BRGM, UJV).</li> </ul> Solid-liquid interface investigation: <ul style="list-style-type: none"> <li>○ Analysis of changes in mineralogical composition and porosity of cement surface layers by surface sensitive techniques.</li> <li>○ Evaluation of the reactions and reaction kinetics.</li> </ul> </li> </ul>			
<b>Main expected outcome per task (corresponding to list above):</b> <ul style="list-style-type: none"> <li>• <b><u>Task 1.1:</u></b> Changes in mineralogy as function of time.</li> <li>• <b><u>Task 1.2:</u></b> Initial diffusion constants and changes of diffusion constants with reaction time (kinetics).  Diffusion constants/ retardation of radionuclides.</li> </ul>			
<b>Description of work to be performed (corresponding to list above):</b> <ul style="list-style-type: none"> <li>• <b><u>Task 1.1:</u></b> Selection of standard low pH materials according CEM II/A-D or CEM III. Preparation and interaction experiments with bentonite water, analytics Construction of diffusion cells, set-up of experiments, pumps, filters, sampling, XRD, SEM for identification of the initial states.</li> <li>• <b><u>Task 1.2:</u></b> Diffusion experiments, regular analysis of pH, water composition, application of HTO tracer for determination of diffusion coefficients ( afterwards exchange of solutions). Application of RN tracers. Post-mortem analysis of the concrete samples (SEM, XRD, autoradiography), porosity measurements, time-dependent surface investigations of batch samples.</li> </ul>			
<b>Cooperations:</b> <ul style="list-style-type: none"> <li>• <i>BRGM, PSI: preparation of specific cement phases for comparison reasons</i></li> </ul>			

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<b>WP 1</b>			<b>KIT</b>
<i>AMPHOS, UJV diffusion studies with some radionuclides.</i> <ul style="list-style-type: none"> <li><i>KIT will make use of results obtained in previous projects such as PEPS, DOPAS</i></li> </ul>			
<b>Added value of Cebama:</b> KIT will derive rate constants indicating the change of mineralogy, porosity, and diffusion constants of low pH concrete in contact with clay water.			
<b>Planned training activities (if any):</b>			
<b>Plans for dissemination:</b> <ul style="list-style-type: none"> <li>Work will be part of PhD thesis. Results will be published in peer reviewed journals.</li> </ul>			

## Appendix to Cebama proposal

## 3 / BRGM

WP 1			BRGM
<b>Overall Objectives:</b> BRGM will perform characterization of Clay/cement materials interfaces. The combined evolution of the mineralogy and microstructure in both materials will be quantify and spatialize in order to implement the data in model to describe the transport properties evolution of the different materials. This work will be conducted in close collaboration with SCK-CEN as the studied clay/cement interfaces (either coming from their underground lab or realized in the lab during this project) will be provided by them			
<b>Objectives for different specific steps/periods (if applicable):</b> <ul style="list-style-type: none"> <li>The common objective of SCK-SCEN and BRGM is to characterize the mineralogical and microstructural changes at the interface of Boom Clay and concrete using a multi-scale investigation and to determine changes in the transport parameters at different time scales (short term lab, long-term anthropogenic analogues). The data obtained within this WP will be tested against the outcome of coupled geochemical-microstructural modeling (WP3)</li> <li>The first step is to characterize the mineralogy and outline the influence of some geochemical perturbations (carbonation, alkaline plume) to constrain the influence of such chemical gradient on the evolution of the mineralogy</li> <li>A second step will be dedicated on the characterization of the microstructure and quantify the evolution of the pore size distribution (PSD) related to change of the mineralogy in the materials at the interface.</li> </ul>			
<b>Main expected outcome:</b> <ul style="list-style-type: none"> <li>Microstructural and mineralogical data on Boom Clay/concrete interfaces together with transport parameters at different time scales and with different concrete compositions.               <ul style="list-style-type: none"> <li>Mineralogy in both cement based materials and clay surrounding formation at the interface allows better constraining the geochemical transport modeling</li> <li>Microstructural data of the materials at the interface to provide 3D PSD in order to perform transport simulation into real 3D pore network.</li> </ul> </li> </ul>			
<b>Description of work to be performed:</b> Using a multi-scale investigation, we aim to identify how Ca-leaching and carbonation (Boom clay pore water contains 14 mmol/L of bicarbonate) change the microstructure at the clay-concrete interface (clogging or opening of pores) and how this alters the transport parameters of the cementitious component. Following types of experiments are proposed: <ul style="list-style-type: none"> <li>Study of the interface Boom Clay/concrete from samples taken from the HADES underground research facility (URF) (Mol, Belgium). Samples will be taken from the Connecting Gallery, in which low-permeability high-pH concrete wedgeblocks are in contact with Boom Clay for 12 years. Several samples could be taken with the intention to determine transport parameters and to perform <math>\mu</math>-structural analysis both on the host rock side and the concrete side.</li> <li>Study of the interface Boom Clay/cement by means of lab experiments with low-strength high-</li> </ul>			

## Appendix to Cebama proposal

WP 1			BRGM
<p>porosity high-pH cement mixture representative for the backfill composition in the Belgian disposal concept (NRVB-type). In these experiments, the chemical conditions can be rigorously controlled.</p> <ul style="list-style-type: none"> <li>• Percolation type experiment: Boom Clay plug in contact with cement plug percolated with pore water. In these experiments, the conditions in the HADES URF can be mimicked (advective flow regime from the clay to the cement). A set of experiments will be set up which can be stopped as function of interaction time. For each interaction time, 2 experiments are performed in parallel, one will be used for measuring the diffusive parameters (by using radiotracers or dissolved gas – to be decided), the other one is foreseen to conduct chemical and <math>\mu</math>-structural analysis.</li> <li>• For both types of experiments we aim for a thorough characterization of the used materials at the beginning of the experiment and respectively after one and two years of interaction.</li> </ul> <p>The analysis of all the samples will be carried out by means of a multi-scale investigation to identify any evidence of perturbation in the cement-based materials and the clay. Chemical, mineralogical and <math>\mu</math>-structural measurements will be performed. For this multi-scale investigation the work will be carried out jointly with SCK-CEN. Samples will be selected based on the outcome of the lab experiments and after first screening using computer tomography. The most detailed analysis will be performed first on the sample from HADES URL and then on the samples from the percolation experiments.</p> <p>SCK•CEN will focus mainly on a) the effect of perturbation at the interface on global transport properties (permeability, gas diffusivity, specific surface area with MIP/N<sub>2</sub> adsorption), b) follow-up of the pore water composition and cation exchange occupancy (ion chromatography), c) limited mineralogical/textural analysis using SEM-EDX, TGA and (quantitative) XRD. Additional mineralogical characterization techniques already applied and coupled on clay/cementitious materials in several studies [1-5], (Infra-Red (IR) Raman spectroscopy coupled to SEM, High-Resolution Solid-State Nuclear Magnetic Resonance (NMR), Transmission Electron Microscopy (TEM) Electron Probe Micro Analyses (EPMA), and micro X-Ray Fluorescence (<math>\mu</math>XRF) will be performed at BRGM.</p> <p>Using an integrated downscaling approach, we aim to quantify and spatialize how the geochemical perturbation surrounding those interfaces will modify the microstructure of both engineered and natural barriers and therefore the effect on the transport properties. A multi-scale investigation methodology that has been developed on clay materials during the European FP7 CATCLAY project will be used to retrieve the <math>\mu</math>-structural changes. The methodology integrates several bulk macroscopic characterization techniques and imaging method to display quantitative data from macroscopic to nanoscopic scale. The association of several 2D/3D techniques (mineral cartography, autoradiography, <math>\mu</math>Tomography-RX, SEM, FIB-nT and TEM) allow to reach a quantitative and spatial distribution of the mineralogy and the pore network, from nanometer to micrometer. Therefore, the influence of the mineralogical and chemical changes on the microstructure will be obtained by: a) 2D quantitative spatial distribution of the mineralogy and its evolution with the effect of the perturbation, b) 2D quantitative spatial distribution of the porosity to localize spatial heterogeneities and porosity evolution as regard to the interface, c) focus on localized area to reach 3D pore network.</p> <p>This robust integrated methodology allows reaching the coherence of several geometric key parameters such as the pore size distribution and the associated mineral distributions. These quantitative data will be used to support lattice Boltzmann simulation to model the diffusion of radionuclides from the pore scale to the pore network.</p>			

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WP 1			BRGM
<p>[1] Lerouge C., Claret F., Tournassat C., Grangeon S., Gaboreau S., Boyer B., Borschnek D., Linard Y. (2014). Constraints from sulfur isotopes on the origin of gypsum at concrete/claystone interfaces. Physics and Chemistry of the Earth, Parts A/B/C, In Press.</p> <p>[2] Jenni A., Mäder U., Lerouge C., Gaboreau S., Schwyn B. (2014). In situ interaction between different concretes and Opalinus Clay Original Research Article Physics and Chemistry of the Earth, Parts A/B/C, In Press.</p> <p>[3] Grangeon S., Claret F., Lerouge C., Warmont F., Sato T., Anraku S., Numako C., Linard Y., Lanson B. (2013). On the nature of structural disorder in calcium silicate hydrates with a calcium/silicon ratio similar to tobermorite. Cement and Concrete Research, Volume 52, October 2013, Pages 31-37.</p> <p>[4] Gaboreau, S., D. Prêt, E. Tinseau, F. Claret, D. Pellegrini, and D. Stammose, 15 years of in situ cement–argillite interaction from Tournemire URL: Characterisation of the multi-scale spatial heterogeneities of pore space evolution. Applied Geochemistry, 2011. 26(12): p. 2159-2171.</p> <p>[5] Gaboreau S., Lerouge C., Dewonck S., Linard Y., Bourbon X., Fialips C.I., Mazurier A., Pret D., Borschnek D., Montouillout V. et al (2012). In-situ interaction of cement paste and shotcrete with claystones in a deep disposal context. American Journal of Science 312, 3 (2012) 314-356.</p>			
<p><b>Cooperations:</b></p> <ul style="list-style-type: none"> <li>• <i>SCK-CEN will provide analogue cement based materials/boom clay interfaces which experienced more than 10 years of interactions and more constrain percolation transport experiences Lattice Boltzmann simulation will be performed by SCK-CEN from the PSD extract from the characterization of the interface obtained by the BRGM team.</i></li> <li>• <i>Cebama partners</i></li> </ul>			
<p><b>Added value of Cebama:</b> multidisciplinary co-operation on international level between specialised research teams will result in an efficient increase in understanding of phenomena playing on clay/concrete interface. In these experiments we will combine the expertise of SCK•CEN regarding percolation-type transport experiments, the availability of analogue samples in the HADES underground research facility (Euridice, Mol, Belgium) and the expertise of BRGM regarding mineralogical and textural analysis in order to come to a coherent evaluation of the perturbations at the clay/concrete interface.</p>			
<p><b>Plans for dissemination:</b></p> <ul style="list-style-type: none"> <li>• Joint papers, posters/presentations at conferences, open research reports ...</li> </ul>			



## Appendix to Cebama proposal

## 4 / NERC-BGS

WP 1			NERC-BGS
<b>Overall Objectives:</b> To understand the long-term impact of low alkali fluids on seal integrity within a radioactive waste repository.			
<b>Objectives for different specific steps/periods (if applicable):</b> <ul style="list-style-type: none"> <li>Examine the interaction of low-alkali cement leachate on bentonite seal performance with specific emphasis on the evolution of strength, permeability and exchangeable cation properties of key system interfaces (in particular interaction between seal, host-rock and canister).</li> </ul>			
<b>Main expected outcome:</b> <ul style="list-style-type: none"> <li>Improved process understanding relating to gallery/deposition hole seal performance and the impact of changing geochemical and stress environments on seal integrity.</li> <li>Understand the rates of reaction/interaction and their impact on strength and permeability of seal components. Identify and quantify key processes that occur and reduce uncertainty of long-term seal integrity and performance for inclusion in future predictive numerical codes.</li> </ul>			
<b>Description of work to be performed:</b> <ul style="list-style-type: none"> <li>The weakest part of any gallery or deposition hole seal will be at the interface between the sealing components and the host rock. A single seal completion may comprise a number of components reflecting different design criteria in order to address specific engineering needs associated with changes in geochemistry and stress. The interaction of these components with the host rock, their evolution in terms of strength/bonding, cation exchange behavior and interfacial permeability, and the sensitivity of these properties to an evolving geochemical and physical environment will be key factors in determining long-term seal performance. In the French repository concept, low-alkali cement is proposed as a mechanical support for bentonite seals as they slowly hydrate to isolate sections of the repository system. The interaction between the cement, bentonite and host rock must be understood in order to inform performance assessment. As such, a matrix of bespoke tests using state-of-the-art experimental systems capable of simulating key repository conditions is proposed.</li> <li>Particular emphasis will be placed on defining the temporal evolution of each interface to changes in geochemistry, mineralogy and stress to assess their impact on the development of permeability (gas and water) and strength (shear strength). Diagnostic tests will be performed in a number of heavy duty shear rigs (Figure X) providing real-time data on the hydromechanical and transport behavior of each interface as it evolves in time and space. This information (including shear strength, fracture transmissivity (gas and water), volume change, normal and shear stresses etc) will be combined with post-test petrological and geochemical analyses (including high-resolution CT scanning) to develop a conceptual model describing seal behavior. This model will be</li> </ul>			

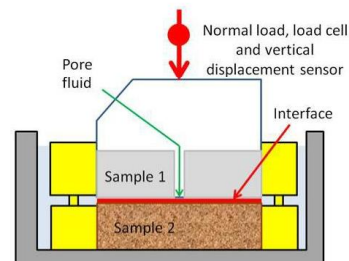


Figure X Bespoke BGS experimental system designed to examine interface behavior

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WP 1			NERC-BGS
subsequently used by Andra to guide and verify future predictive numerical models aimed at repository performance assessment.			
<b>Cooperations:</b> <ul style="list-style-type: none"> <li><i>This laboratory-based task is proposed in consultation with Andra and will compliment proposed field-scale experiments to be undertaken at Bure. Test material will be provided by Andra who will liaise closely with researchers at BGS to define the matrix of tests.</i></li> <li><i>Outputs from this study are of direct relevance to other WMO programmes including Ondraf-Niras, COVRA and potentially SKB, POSIVA and the RWML.</i></li> </ul>			
<b>Added value of Cebama:</b> Fundamental enhanced process understanding of the performance of seals in a low-alkali environment, to inform the subsequent programme of work (in particular WP3). BGS will provide world class expertise in this field and will compliment field-scale experiments undertaken at BURE all of which will increase knowledge and confidence on seal integrity and performance in a repository.			
<b>Planned training activities (if any):</b> <p>Work will be undertaken by experienced researchers within the BGS who have a proven track record in science delivery. Experimental activities will utilize existing techniques thereby reducing 'risk' of non-delivery potentially associated with any experimental activity.</p>			
<b>Plans for dissemination:</b> <p>Through Work Package 4, which focusses on dissemination and knowledge transfer, we propose to disseminate, interact and engage with stakeholders. These are important facets of this task together with regular meetings with Andra and other interested WMO's will be undertaken. In addition, BGS scientists will actively participate in knowledge transfer with the wider academic community through proceedings/workshops, and will submit at least two peer-reviewed publications during the course of the project (in conjunction with WP4). Annual progress statements will also be produced for dissemination by project participants and collaborating WMO's.</p>			

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## 5 / CIEMAT

WP 1			CIEMAT
<b>Overall Objectives:</b>  Objective 1: To investigate the hydro-geochemical interactions of high pH concrete (OPC)/FEBEX bentonite and high pH concrete (OPC)/Grimsel granite interfaces in large scale FEBEX in situ test and high pH concrete (OPC)/FEBEX bentonite interface in HB6 cell long-term experiment.  Objective 2: Quantification of porosity change phenomena affecting transport due to mineralogical alteration in the several materials involved in the in situ and laboratory experiments and extent of the alteration of physicochemical properties of these materials.			
<b>Objectives for different tasks (<i>specific to your organization</i>):</b>  <ul style="list-style-type: none"> <li>• <b><u>Task 1.1: Establishing known State-of-the-Art, defining boundary conditions for experimental studies and details of analytical methods for sample characterization</u></b> <ul style="list-style-type: none"> <li>- Defining the sampling and conditioning requirements of the in situ large scale interfaces (FEBEX) (month 1 to 6).</li> <li>- Definition of the procedures for hydrogeochemical and transport characterization of in situ and laboratory experiments (month 1 to 6).</li> </ul> </li> <li>• <b><u>Task 1.2: Characterization &amp; experimental studies.</u></b></li> <li>• <b><u>1.2.2: Interfaces with bentonite</u></b> <ul style="list-style-type: none"> <li>- Characterization of OPC/FEBEX bentonite and OPC/granite interfaces to identify parameters and processes taken place during the evolution of the EBS in a long-term in situ real scale test (month 6 to 42).</li> <li>- Dismantling and characterization of HB6 cell (OPC/FEBEX bentonite interface) after 10 years of operation to identify geochemical processes affecting transport in a long-term laboratory scale test (month 12 to 38).</li> <li>- Obtaining data on OPC porosity under different chemical conditions (month 6 to 42).</li> <li>- Data interpretation will be based on the up-scaling (spatial and temporal) of geochemical processes and transport phenomena at the interface scale and the extent of the altered zone. For this, integration and comparison among laboratory (HB) and in situ (FEBEX) experiments at different temporal and spatial scales will be made and conclusions for PA will be obtained (month 38 to 48).</li> </ul> </li> </ul>			
<b>Main expected outcome per task (corresponding to list above):</b>  <ul style="list-style-type: none"> <li>• <b><u>Task 1.1:</u></b> <ul style="list-style-type: none"> <li>- Establishing, according to the previous knowledge, of sub-sampling conditions and characterization of both in situ and experimental interfaces.</li> </ul> </li> <li>• <b><u>Task 1.2:</u></b> <ul style="list-style-type: none"> <li>- Hydrogeochemical evolution of the FEBEX site, at interface and larger scale, after 18 years of host rock (granite)/bentonite/concrete interaction, with emphasis in the alkaline plume extension.</li> <li>- Assessment of the impact on primary safety functions of EBSs of concrete interface effects, predicted by previous investigations, in terms of geochemical processes taken place during the early stage evolution of the EBS at mm to cm scale.</li> </ul> </li> </ul>			

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WP 1			CIEMAT
<ul style="list-style-type: none"> <li>- OPC porosity changes due to chemical perturbations and its effects on the radionuclide (RN) transport.</li> <li>- Precise establishment of the porosity change affecting transport in the several materials involved. Up-scaling (spatial and temporal) of geochemical processes and transport phenomena at the interface scale, paying attention to those affecting porosity, as well as determination of the extension of the altered zone, with emphasis in the alkaline plume.</li> </ul>			
<p><b>Description of work to be performed (corresponding to list above):</b></p> <p>Two different groups of CIEMAT are involved in the proposed work: the Applied Environmental Geology Unit (CIEMAT-AEG) and the Physico-Chemistry of Actinides and Fission Products Unit (CIEMAT-PAFP). The description of task work is organized considering the specific contribution of each group.</p> <ul style="list-style-type: none"> <li>• <u>Task 1.1</u></li> </ul> <p>The Applied Environmental Geology Unit of Ciemat (CIEMAT-AEG) has been involved in the FEBEX experiment at the Grimsel Test Site from the beginning in 1997. The experiment will be dismantled in the near future (2015), after 18 years of continuous heating and natural re-saturation under partly aerobic conditions, and samples (concrete/bentonite and concrete/granite interfaces) will be retrieved during the foreseen dismantling (FEBEX-DP) and will be analyzed. The test would provide a unique opportunity for checking the evolution and state of the materials under conditions very close to the real ones during operation with respect to humidity, stresses, chemical environment and temperature.</p> <p>Additionally, the CIEMAT-AEG accounts in their laboratories with one experiment initiated during NF-PRO EU Project: HB6 cell, a small scale laboratory experiment with high pH concrete and FEBEX bentonite, which should be dismantled during the project, after 10 years of operation. The valuable information from previous NF-PRO and PEBS projects regarding processes occurring at interface scale after sequential (in terms of time) dismantling of HB-type cells will be available to establish the temporal evolution of geochemical processes at the interface. Therefore, the participation in projects NF-PRO and PEBS provides a valuable background on the dismantling, preservation and conditioning of samples for the interface studies.</p> <p>The specific work of CIEMAT-AEG on this task, in collaboration with UAM and CSIC would include:</p> <ul style="list-style-type: none"> <li>- Conditioning of the samples (pieces of the different materials and/or cores including the interfaces) coming from the in situ experiment.</li> <li>- Dismantling, sampling and conditioning of the samples coming from the laboratory experiments.</li> <li>- Decisions on characterization procedures of the experiments.</li> </ul> <ul style="list-style-type: none"> <li>• <u>Task 1.2</u></li> </ul> <p>CIEMAT-AEG work for this task would include: the hydrogeochemical characterization of samples coming from the FEBEX and HB6 experiments; a detailed characterization of physical and physical-chemical parameters and the study of geochemical interface processes and transport phenomena. Also, as a macroscopic assessment of the changes observed in the concrete, the gas and water permeability of some samples will be determined.</p> <p>The specific work on geochemical processes (with emphasis on the extension of the alkaline plume) and transport phenomena would include:</p> <ul style="list-style-type: none"> <li>- Integration of hydro-geochemical results of the FEBEX in situ experiment (experiment</li> </ul>			

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WP 1			CIEMAT
<p>scale) coming from annual sampling campaigns (since 2003) of boreholes surrounding the experiment.</p> <ul style="list-style-type: none"> <li>- Determination of natural chemical tracers as stable isotopes in carbonates or the content of several trace elements to interpret geochemical processes in the high pH concrete/FEBEX bentonite and high pH concrete/Grimsel granite interfaces.</li> <li>- Chemical analysis of extractable soluble salts of the EBS materials and bentonite exchangeable cations in the interface of the two proposed experiments in order to have indicators of major elements chemistry involved in mass transport (interface scale).</li> <li>- Analysis of mineralogy of the EBS materials and bentonite in the interface of the two proposed materials in order check mineralogical alteration processes related to the interface zone affecting also mass transport.</li> <li>- Determination of pore size distribution (MIP), specific surface area (BET), and gas and water permeability of specific samples.</li> </ul> <p>The Physico-Chemistry of Actinides and Fission Products Unit of Ciemat (CIEMAT-PAFP) will perform detailed studies related to the porosity changes in OPC associated to chemical perturbations, with special focus on their possible effects on RN transport. The impact on solid porosity of groundwater changes in: pH, redox, sulphates, carbonates and calcium will be analysed. All the tests will be performed under controlled N<sub>2</sub>+CO<sub>2</sub> atmosphere.</p> <p>Through-Diffusion tests with conservative elements (HTO and Cl<sup>-</sup>), will be initially carried out using a synthetic water in equilibrium with the solid to determine the “initial” porosity for the neutral (HTO) and anionic (Cl<sup>-</sup>) species. After this first stage, the chemical composition of the contacting water will be modified and the effects of the modifications of the abovementioned parameters on the solid matrix analysed. Different samples will be used for studying each parameter. In particular, experiments with HTO (and Cl<sup>-</sup>) will be periodically performed to kinetically analyse porosity changes. The study will be completed performing diffusion tests with non-conservative elements (Cs, Sr and Se/Tc) in the initial and modified samples. The post-mortem analysis of some samples is also foreseen, including ion beam techniques analyses (RBS and uPIXE), microscopy, BET, XRD.</p>			
<p><b>Cooperations:</b></p> <p><b>CEBAMA internal partners</b></p> <ul style="list-style-type: none"> <li>• <b>CIEMAT-AEG/UAM/CSIC (within objective 1):</b> Selection of the more suitable cores of the large-scale test will be first performed by collaboration among CIEMAT-AEG, UAM and CSIC. The CSIC group will hold the task of the characterization of aging of concrete cores for profile microstructural changes, from host rock to bentonite, of concrete components (cement paste and aggregate). CIEMAT-AEG will deal with geochemical and petrophysical data at a larger scale, considering the different materials involved in the experiments and UAM with the characterization of interfaces and mineralogical alteration. The integration of CIEMAT-AEG/ UAM /CSIC will try to answer how chemical interactions affect transport properties at several interfaces of interest in CEBAMA.</li> <li>• <b>CIEMAT-AEG/UAM/CSIC (within objective 2):</b> CSIC will design the OPC concrete used in FEBEX experiment. They will also characterize the concrete components alteration near or at the interface focusing to the ageing of concrete after the infiltration test with granitic ground water (from BO-ADUS Borehole) and effect in different transport properties including porosity. Besides CSIC will design the ad-hoc low-pH type of concrete in function of the environmental scenario, for studding pore granitic ground water in the aging and transport properties loss of concrete. Different concrete samples and initial concrete leached waters will be distributed to</li> </ul>			

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WP 1			CIEMAT
<p>CIEMAT-AEG and UAM for interface experiments and analysis.</p> <ul style="list-style-type: none"> <li>• <b>CIEMAT-AEG/UAM/CSIC</b> will collaborate with UDC to provide input for modeling from the HB laboratory experiments.</li> </ul> <p><b>CEBAMA external partners</b></p> <ul style="list-style-type: none"> <li>• ENRESA will be interested in founding part of PhD training of laboratory concrete experiments in contact with granitic waters affecting bentonite, including short-term infiltration tests and the up-scaling to long term HB cell experiments performed and hosted at the CIEMAT facilities.</li> </ul>			
<p><b>Added value of Cebama:</b></p> <p>Analysis of hydro-geochemical processes and physicochemical properties of EBSs in large-scale in situ experiments and its comparison (upscaling) with laboratory tests in the collaborative framework of expert groups in that specific working field.</p>			
<p><b>Planned training activities (if any):</b> experiments support and cooperation to the development of the PhD planned by UAM/CSIC in aspects related to the alteration of concrete/bentonite interface.</p>			
<p><b>Plans for dissemination:</b></p> <p>Research outcome will be published in CIEMAT Technical Reports, relevant peer reviewed journals and presented at topic related conferences and meetings.</p>			

## Appendix to Cebama proposal

## 6 / TU Delft

WP 1			TU Delft
<b>Overall Objectives:</b> <ul style="list-style-type: none"> <li>Investigate pore evolution and the impact of processes at the interface between Boom clay and high-pH concrete on chemical and physical properties, in relation to Dutch and Belgian disposal concepts.</li> <li>Propose theoretical models for designing the experiments and interpretation of the obtained results (in collaboration with NRG in Petten).</li> </ul>			
<b>Objectives for different tasks (specific to your organization):</b> <ul style="list-style-type: none"> <li><b><u>Task 1.1: Establishing known State-of-the-Art, defining boundary conditions for experimental studies and details of analytical methods for sample characterization</u></b> Duration: 2 PM</li> <li><b><u>Task 1.2: Characterization &amp; experimental studies.</u></b> <ul style="list-style-type: none"> <li>Carry out laboratory scale experiments on the interaction at the interface between the Boom clay and concrete with the mechanical support function (composition specified in Table 1), and the influence of such interaction on the pore evolution at the interface. The concrete and clay materials are specific for the Dutch case [1, 2] and will be provided by COVRA. The primary choice of samples will be presented by unoxidized Boom clay and fresh but fully set concrete (2-3 months after the preparation).</li> <li>Investigate transport properties of Ca, Mg, C and S through the interface using electromigration cell as an acceleration method.</li> <li>Determine diffusion profiles of Ca, Mg, C and S and water movement in clay/concrete samples using neutron activation analysis and tracers (S-35, T, D) as the function of time and the applied voltage.</li> <li>Use X-ray imaging technique for the visualization and reconstruction of pores. Depending on the results, positron annihilation technique can be applied for characterization of nanoporosity.</li> </ul>  Duration: 12 PM </li> <li><b><u>Task 1.3 Conceptual modelling and data interpretation.</u></b>  Interpretation of the results obtained in Task 1.2 and using of this data as an input for developing and testing state-of-the art numerical model for processes at cement-clay interface (in collaboration with CEBAMA partner from WP3 - NRG).  Duration: 3 PM</li> </ul>			
<b>Main expected outcome per task (corresponding to list above):</b> <ul style="list-style-type: none"> <li><b><u>Task 1.1:</u></b> Literature review</li> <li><b><u>Task 1.2:</u></b> Experimental data on the pore structure at the interfaces (X-ray scanning, positron annihilation) and the evolution of pores due to the diffusion of macroelements Ca, Mg, C, S.</li> </ul>			

## Appendix to Cebama proposal

WP 1			TU Delft		
<ul style="list-style-type: none"><li><u>Task 1.3:</u> Processed data to be used as an input for WP3.</li></ul>					
<b>Description of work to be performed (corresponding to list above):</b>					
Before the start of intended project activities at TU Delft (January-February 2016) 2 PM during the first year will be dedicated to testing the experimental facilities and preparation of some clay/concrete samples.					
1. <u>Task 1.1:</u>					
Collecting information available in the literature, planning and preparing for the experiments.					
2. <u>Task 1.2:</u>					
In the recent report of the Dutch waste management organization COVRA several types of cements were suggested depending on their different functions [2]:					
3. Portland fly ash unreinforced cement for concrete gallery lining (mechanical support) (Table 1),					
4. foam concrete as a backfill material (Table 2),					
5. rheoplastic concrete (RPC) and self-compacting concrete (SCC) as part of a waste package (waste containment) (Table 3).					
<b>Table 1. Composition mechanical support (concrete in reinforced concrete segments)</b>					
Component/parameter	Type				
Cement	CEM II/A to B-(V)	386	kg m <sup>-3</sup>		
Water		125	kg m <sup>-3</sup>		
Plasticiser	Woermann BV 514	1.33	kg m <sup>-3</sup>		
Superplasticiser	Woermann FM 30	3.65	kg m <sup>-3</sup>		
Fine aggregate	quartz sand: 0-2 mm	615	kg m <sup>-3</sup>		
Coarse aggregate	quartz gravel: 2-8 mm	612	kg m <sup>-3</sup>		
Coarse aggregate	quartz gravel: 8-16 mm	700	kg m <sup>-3</sup>		
w/c	Property	0.39			
<b>Table 2. Composition enclosure emplaced waste (backfill – foam concrete)</b>					
Component	Receipt for 1 m <sup>3</sup> of Aercrete FC 1200 to 1600 kg m <sup>-3</sup>	Type for OPERA	1200 kg m <sup>-3</sup>	1600 kg m <sup>-3</sup>	
Cement	360 to 400 kg	CEM I	360	400	kg m <sup>-3</sup>
Water	140 to 160 kg	-	140	160	kg m <sup>-3</sup>
Fine aggregate	750 to 1100 kg	Quartz sand: 0-4 mm	750	1100	kg m <sup>-3</sup>
Foaming agent Synthetic surfactant	0.57 to 0.36 l	Foaming agent TM 80/23 Synthetic	1	1	kg m <sup>-3</sup>
Water	21.3 to 13.6 l	Water	21.3	13.6	kg m <sup>-3</sup>
Air	434 to 277 l	Air	0	0	kg m <sup>-3</sup>
w/c		property	0.45	0.43	



## Appendix to Cebama proposal

WP 1			TU Delft
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Table 3. Composition containment of waste (RPC-HLW)

Component/ parameter	Type		
Cement	CEM I/42.5 N HS LA (LH)	350	kg m <sup>-3</sup>
Water	-	175	kg m <sup>-3</sup>
Filler	Calcitec 2001 ME	50	kg m <sup>-3</sup>
Plasticiser Polycarboxylic ether based	Glenium 27/20	4.41	kg m <sup>-3</sup>
Fine aggregate	Limestone: 0-4 mm	708	kg m <sup>-3</sup>
Coarse aggregate	Limestone: 2-6 mm	414	kg m <sup>-3</sup>
Coarse aggregate	Limestone: 6-14 mm	191	kg m <sup>-3</sup>
Coarse aggregate	Limestone: 6-20 mm	465	kg m <sup>-3</sup>
w/c	Property	0.50	

N Usual initial strength, HS High Sulphate resistance, LA Low Alkali content , (LH Low Hydration heat)

Current research proposal will be focused on the study of processes at the interface between high-pH cementitious material served as a mechanical support (Table 1) and the unoxidized Boom clay. This type of concrete will have a direct contact with the surrounding clay in the repository conditions. At later stages the other two types of concrete might be also involved and tested (depending on the experimental time available).

The experimental part of the project will consist of two tasks:

5.2.1. Studies of the diffusive transport of macro elements (Ca, Mg, S, C) through the interfaces based on the nuclide transport experiments.

5.2.1.1. The major focus will be on the characterization of behaviour of calcium, magnesium, carbon (in the form of carbonate) and sulphur, which are the elements responsible for changes in physical properties (pore blocking, calcite precipitation, sulphate attack etc.). Their accumulation/dissolution near the interface will be studied and kinetic models will be developed. The migration will be accelerated by using an electro diffusion cell. Acceleration of the process is the major advantage of the technique [3]. The applicability of electro migration for cement systems was demonstrated in several papers [4, 5]. In the current research project we will investigate kinetic parameters of the processes as a function of concentration, pH, electric potential.

5.2.1.2. Characterization of water mobility in the pores will be performed using neutron imaging with the help of deuterium water (or tritium-labeled water). In this way, the diffusion properties of water in clay and cement and the permeability can be estimated. In combination with nuclide transport data this will give better understanding of diffusion and advection processes at interfaces.

5.2.1.3. Characterization of concentration profiles of stable elements at the interfaces is essential for monitoring changes that could influence the pore structure in the long term.

5.2.2. Analysis of pore structure at interfaces. X-ray imaging (at microscale) will be used for

## Appendix to Cebama proposal

WP 1			TU Delft
<p>visualization of liquid flow through porous samples, reconstruction and texture quantifications. High-resolution X-ray imaging system is available at RID equipped with a high-power X-ray tube (up to 300kV) to identify certain sample characteristics such as size and location of voids. Depending on the results obtained by X-ray imaging, a positron annihilation technique can be applied with POSH-PALS and POSH 2D-ACAR instruments for characterization of free volume in materials at nanoscale (both bulk samples and interfaces).</p> <ul style="list-style-type: none"> <li>• <b>Task 1.3:</b> Analysis of the data obtained in Task 1.2. Preparing a scientific report.</li> </ul> <p>All the experiments will be performed at the laboratory scale. The obtained data will be used for underpinning of large scale scenarios for the Safety Case.</p> <p>References:</p> <ol style="list-style-type: none"> <li>6. E. Verhoef, E. Neeft, J. Grupa, A. Poley, Outline of a disposal concept in clay, OPERA-PG-COV008 (2011) 1-17.</li> <li>7. E.V. Verhoef, A.M.G. de Bruin, R.B. Wiegiers et al., Cementitious materials in OPERA disposal concept in Boom Clay, OPERA-PG-COV020 (2014) 1-17.</li> <li>8. N. Maes, H. Moors, A. Dierckx et al., The assessment of electromigration as a new technique to study diffusion of radionuclides in clayey soils. Journal of Contaminant Hydrology 36 (1999) 231–247.</li> <li>9. F. Frizon, S. Lorente, C. Auzuech, Nuclear decontamination of cementitious materials by electrokinetics: an experimental study, Cement and Concrete Research 35 (2005) 2018 – 2025.</li> <li>10. X. He, X. Shi, Chloride Permeability and microstructure of portland cement mortars incorporating nanomaterials, Transportation Research Record: Journal of the Transportation Research Board, No. 2070 (2008) pp. 13–21.</li> </ol>			
<p><b>Cooperations:</b></p> <ul style="list-style-type: none"> <li>• Close collaboration with Cebama partner NRG (WP3).</li> </ul>			
<p><b>Added value of Cebama:</b></p> <ul style="list-style-type: none"> <li>• Improved understanding of processes at clay/concrete interfaces (also specific to the Dutch case).</li> <li>• Unique insights of the problem obtained by the neutron- and positron-based techniques available at the RID of TU Delft.</li> <li>• Combination of experimental results to be obtained at RID with a sound expertise of NRG in using the ORCHESTRA code for modelling reactive transport in concrete will contribute to better understanding of the C/Ca behaviour/pore evolution at different clay/concrete interfaces.</li> <li>• Interpretation of the results on the basis of cooperation between TU Delft and NRG.</li> <li>• Education of specialists in the field of nuclear waste disposal.</li> </ul>			
<p><b>Planned training activities (if any):</b></p> <ul style="list-style-type: none"> <li>• The research fellow will be trained to work safely with radiation and is expected to complete a radiation safety course, for which he/she will receive the (inter)nationally recognized certificate Health Physics level 5B or level 3.</li> <li>• Training students in using reactive transport model as part of TU-Delft curriculum.</li> <li>• ORCHESTRA geochemical modelling and transport course.</li> </ul>			

### Appendix to Cebama proposal

WP 1			TU Delft
<b>Plans for dissemination:</b> <ul style="list-style-type: none"> <li>• Use of the materials in lectures for students within the specialization Nuclear Science and Engineering.</li> <li>• Publication of the results in peer-reviewed scientific journals.</li> <li>• Distribution among OPERA partners in the Netherlands and in Belgium, and among the Cebama partners.</li> <li>• Use of the material as a starting point for future research projects in the NL and in Europe.</li> </ul>			

## Appendix to Cebama proposal

## 10 / RWMC

WP 1			RWMC
<b>Overall Objectives:</b> 1. To get the realistic data for the expansion of altered zone around the contact between cementitious and bentonitic materials.  2. To obtain the fundamental data of change of mechanical properties arose from the chemical alteration. Because mechanical and Hydraulic characteristics of bentonitic materials are modeled as functions of effective montmorillonite density, i.e. content of montmorillonite and dry-density of that material, dissolution and/or precipitation of montmorillonite and secondary minerals are important data.  To develop the method of H-M-C coupled calculation for mainly bentonitic material. The main point of RWMC's work is the reciprocal influences between change of (1) mechanical and hydraulic properties due to chemical evolution and (2) changes of the chemical alteration due to the change of hydraulic properties. Those changes due to Cement-Bentonite interaction.  Reference bentonitic material is Kunigel V1 and cementitious materials are OPC and Low alkalinity cement in Japan, but other cementitious materials, i.e. flyash mixed cement, are also in consideration.-			
<b>Objectives for different specific steps/periods (if applicable):</b>  Expected outcome of the bellow will be useful data for the modeling of T-H-M-C coupling calculations in WP3, especially for H-M-C coupling.  The results of WP1 will be used as input for WP3. The outcome of this work could be useful for evaluating the long term performance of barrier system including cement-clay systems.  RWMC has already done the modeling of the alteration of various cementitious materials, i.e. OPC and mixed cement, and also has been carrying out many experiments of cement-bentonite coupling. RWMC has also natural analogue project of pH~11 alkaline alteration of bentonite formation in Philippines. Those results will be able to share in CEBAMA project, and RWMC will reflect the fruits of those experiments and studies, including the dissolution rate of montmorillonite under compacted condition, as the input for the long term mineralogical changes. The changes of mechanical and hydrological properties have been modeled as a function of "effective montmorillonite density" in FY2000 report and 2 <sup>nd</sup> progress report for TRU waste disposal in Japan. RWMC uses that relationship for the M-C coupled calculation.			
<b>Main expected outcome:</b> (1) Thickness of the altered zone of bentonitic or clay of long-term (5 to 10 years or more) immersed specimen by Ca-XAFS experiment (and natural analogue bentonitic formation). That will be a useful to explain the stability of the multi-barrier systems. (2) Generation and growth ratio of secondary zeolitic minerals, i.e. clinoptilolite, illite and analcime, under compacted and alkaline (pH~12.5) condition will be obtained by Phase-Shift-Interferometry and Atomic Force Microscopy. (3) Mechanical properties such as swelling pressure, porosity-compaction relation and so of zeolite-montmorillonite mixture as a model altered bentonitic material (4) Change of swelling pressure of montmorillonite during the change from Na-type into Ca-type-			

## Appendix to Cebama proposal

WP 1			RWMC
<b>Description of work to be performed:</b> <ol style="list-style-type: none"> <li>Ca-XAFS observation of long-term immersed C-B coupled samples and/or artificial analogue specimen. This experiment will be performed till FY2016 and 1 or 2 specimen per year because of the limitation of the machine time of synchrotron-XAFS at KEK (72 to 96hrs/year). Specimen for XAFS analyses will be 5 to 10 years immersed CC-bentonite coupled specimen that is similar to the diffusion test. And if the artificial analogue such as the boring core of concrete-clay contact at URL, for examples the silo of formally GMT in Grimsel, will be demanded from other participant, RWMC may be able to include that to their plan of XAFS experiment.</li> <li>Generation and growth ratio of secondary zeolitic minerals, i.e. clinoptilolite, illite and analcime, under compacted and alkaline (pH~12.5) condition will be obtained by Phase-Shift-Interferometry and Atomic Force Microscopy. In the previous work, RWMC has performed the pseudo in-situ alteration experiment of montmorillonite under the compacted and alkaline condition by using phase-shift interferometry. In that experiment, zeolitic secondary phases such as written above. In this WP1, RWMC will confirm the growth ratio of those secondary minerals.</li> <li>Experiment to obtain the physical properties such as e-logP, i.e. porosity-compaction relation, by using the mixture of montmorillonite and expected secondary minerals (Ex. some types of zeolite). RWMC had already performed such experiment using pure montmorillonite, bentonite (Kunigel V1) and montmorillonite-sand mixture which has the same content of montmorillonite. In that experiments, the mechanical properties of mixture were almost the same as those of bentonite. So that, the mechanical properties of altered bentonite had been expected to be the same as those of bentonite with the same montmorillonite content. In this experiment, RWMC will confirm that the way how to predict the mechanical properties of altered, i.e. containing secondary minerals such as zeolitic ones.</li> <li>Change of swelling pressure of montmorillonite during the change from Na-type into Ca-type will be obtained. RWMC already started some experiments to obtain such changes of mechanical properties in the last fiscal year. In FY2015, RWMC will be able to publish some reports of those experiments.</li> </ol>			
<b>Cooperations</b> <ul style="list-style-type: none"> <li>RWMC may be able to carry out Ca-XAFS observation for the C-B interface of artificial analogue samples or some long term immersion specimen from the other partners.</li> <li>RWMC also has a project of gas-migration mainly in bentonitic material. It may be able to be another item.</li> </ul>			
<b>Plans for dissemination:</b> Results of RWMC's project must be published as annual report to METI, and will be submitted to scientific journals.			

## Appendix to Cebama proposal

## 11 / SCK•CEN

WP 1			SCK•CEN
<b>Overall Objectives:</b>  SCK•CEN will perform experiments in order to characterize the interface Boom Clay/High pH concrete in terms of changes in transport properties and microstructure due to Ca leaching and carbonation. The work will be performed in close cooperation with BRGM who will focus on microstructural characterization techniques.			
<b>Objectives for different tasks (<i>specific to your organization</i>):</b>  <ul style="list-style-type: none"> <li>• <b>Task 1.2: Characterization &amp; experimental studies.</b>  The common objective of SCK•CEN and BRGM is to determine changes in the transport parameters at different time scales (short term lab, long-term anthropogenic analogues) by characterizing the mineralogical and microstructural changes at the interface of Boom Clay and concrete using a multi-scale investigation. The data obtained within this WP will be served to modellers (including those at SCK•CEN) within WP 3 in order to test/validate the outcome of coupled geochemical-microstructural modeling. </li> </ul>			
<b>Main expected outcome per task (corresponding to list above):</b>  <ul style="list-style-type: none"> <li>• <b>Task 1.2:</b> Transport parameters and associated microstructural data on interface Boom Clay/concrete at different time scales and with different concrete compositions. </li> </ul>			
<b>Description of work to be performed (corresponding to list above):</b>  <u>Task 1.2:</u> Using a multi-scale investigation, we aim to identify how Ca-leaching and carbonation (Boom clay pore water contains 14 mmol/L of bicarbonate) alters the transport parameters of the cementitious component and changes the microstructure at the clay-concrete interface (clogging or opening of pores).  Following types of experiments are planned: <ul style="list-style-type: none"> <li>• <b>Study of an existing interface Boom Clay/concrete (Y1&amp;2)</b> from samples taken from the HADES underground research facility (URF) (Mol, Belgium). Samples will be taken from the Connecting Gallery, in which <i>low-permeability high-pH concrete (CEMIII +PFA)</i> wedgeblocks are <i>in contact with Boom Clay for 12 years</i>. Several samples will be taken with the intention to <i>determine transport parameters and to perform <math>\mu</math>-structural analysis</i> both on the host rock side and the concrete side.</li> <li>• <b>Study of the interface Boom Clay/cement</b> by means of lab experiments with <i>low-strength/high-porosity high-pH cement mixture</i> representative for the backfill composition in the Belgian disposal concept (<i>NRVB-type: OPC/limestone flour/hydrated lime</i>). In these experiments, we start from <i>newly manufactured materials</i> and interfaces while rigorously controlling the chemical conditions. <ul style="list-style-type: none"> <li>• <i>Batch type experiments (Y1&amp;2):</i> immersion of a cement plug into a Boom Clay slurry. Several experiments will be set up and at specified times a cement plug is removed <i>for <math>\mu</math>-structural analysis</i>. The <i>chemical composition</i> of the slurry will also be followed up. The water excess in these experiments will cause a more rapid cement degradation than in other types of experiments.</li> <li>• <i>Percolation/column type experiment (Y2-4):</i> a) Boom Clay plug in contact with cement plug percolated with clay pore water, b) Cement plug alone percolated with clay pore water. During percolation, the <i>permeability</i> will be monitored over time and the <i>pore</i></li> </ul> </li> </ul>			

## Appendix to Cebama proposal

WP 1			SCK•CEN
<p><i>water composition</i> will be analysed depending on availability. In these experiments, the conditions in the HADES URF can be mimicked (advective flow regime from the clay to the cement). Sets of experiments will be set up that will allow to characterize the evolution of the microstructure after different interaction times. After each defined interaction time, 2 experiments will be stopped and analyzed. One is foreseen to conduct <i>chemical</i> and <i>μ-structural analysis</i>. The other one will be used for measuring the <i>diffusive parameters by using radiotracers (classical diffusion and/or PET) or dissolved gas</i> – to be decided.</p> <ul style="list-style-type: none"> <li>For both types of experiments we aim for a thorough characterization of the used materials at the beginning of the experiment and respectively after <i>one and two years of interaction</i>.</li> </ul> <p>The analysis of all the samples will be carried out by means of a multi-scale investigation to identify any evidence of perturbation in the cement-based materials and the clay. Chemical, mineralogical and <i>μ-structural</i> measurements will be performed. For this multi-scale investigation the work will be carried out jointly with BRGM (France). Samples will be selected based on the outcome of the lab experiments and after first screening using computer tomography. The most detailed analysis will be performed on the sample from HADES URF and the samples from the percolation experiments.</p> <p>At SCK•CEN we will focus mainly on a) the effect of perturbation at the interface on global transport properties (permeability, gas diffusivity, specific surface area with MIP/N<sub>2</sub> adsorption), b) follow-up of the pore water composition and cation exchange occupancy (ion chromatography), c) limited mineralogical/textural analysis using SEM-EDX, TGA and (quantitative) XRD. The bulk of the mineralogical/textural analyses will be performed by BRGM (we refer to their description of work for more details). HZDR will be involved in analysing transport pathways in fresh and altered concrete samples by means of PET measurements (selection of a few samples).</p> <p>The data gathered will be <i>transferred to our SCK•CEN colleagues working in WP3 who developed a microstructural modeling tool using Lattice-Boltzmann coupled to PHREEQC</i> in order to simulate the effect of chemical degradation processes on the transport properties.</p> <p>We would like to use this model to interpret the experiments described above.</p>			
<p><b>Cooperations:</b></p> <ul style="list-style-type: none"> <li><i>BRGM: microstructural analysis/imaging of samples from interfaces Boom Clay/concrete.</i></li> </ul> <p>1) Additional mineralogical characterization techniques to those cited above (Infra-Red (IR) Raman spectroscopy coupled to SEM, High-Resolution Solid-State Nuclear Magnetic Resonance (NMR), Transmission Electron Microscopy (TEM) Electron Probe Micro Analyses (EPMA), and micro X-Ray Fluorescence (μXRF)).</p> <p>2) A multi-scale investigation methodology that has been developed on clay materials during the European FP7 CATCLAY project will be used to retrieve the <i>μ-structural</i> changes. The methodology integrates several bulk macroscopic characterization techniques and imaging method to display quantitative data from macroscopic to nanoscopic scale. The association of several 2D/3D techniques (mineral cartography, autoradiography, <i>μTomography-RX</i>, SEM, FIB-nT and TEM) allow to reach a quantitative and spatial distribution of the mineralogy and the pore network, from nanometer to micrometer.</p> <ul style="list-style-type: none"> <li><i>HZDR: analysing transport pathways in fresh and altered concrete samples by means of PET measurements.</i></li> </ul>			

### Appendix to Cebama proposal

WP 1			SCK•CEN
<p><b>Added value of Cebama:</b> multidisciplinary co-operation on international level between specialised research teams will result in an efficient increase in understanding of phenomena playing on HR/CC interface. In these experiments we will combine the expertise of SCK•CEN regarding percolation-type transport experiments, the availability of analogue samples in the HADES underground research facility (Euridice, Mol, Belgium), the expertise of BRGM regarding mineralogical and textural analysis and the expertise of HZDR in PET analysis of porous structures in order to come to a coherent evaluation of the perturbations at the clay/concrete interface.</p> <p>The obtained info will be implemented/used directly by ONDRAF/NIRAS in the development of their safety case.</p>			
<p><b>Plans for dissemination:</b></p> <ul style="list-style-type: none"> <li>• Joint papers, posters/presentations at conferences, open research reports...</li> </ul>			



## Appendix to Cebama proposal

## 13 / UJV

WP 1			UJV
<b>Overall Objectives:</b> <p>The objective of the Czech partners (UJV Řež a.s. and CTU) is to contribute to research on influence of mutual interaction between cement based materials (OPC, LPC) and bentonite (Czech Ca-Mg bentonite) on properties of both materials, being present in deep geological repository. Mutual interaction can influence mechanical, rheological and transport properties of both materials.</p> <p>Planned works will include both in-situ and laboratory originated samples, exploiting the possibility to use materials, being under mutual interaction under different conditions within the period from 10 to 60 months. Samples from previous in-situ experiments in Josef gallery will be available (some being aged since 2010). The effects of cement/bentonite interaction on mechanical, rheological, chemical and transport properties under defined temperature will be studied. Use of OPC is envisaged, however LPC cement, provided by VTT will be used for laboratory studies. The tests on the samples are complementary with CTU tests.</p> <p>One of the most important material property, influencing safety function of engineered barrier, is diffusivity. Both total and available diffusivity can be easily quantified using radioactive tracer (<math>^3\text{H}</math>, <math>^{36}\text{Cl}</math>). Moreover, diffusion and its changes for other safety relevant tracers (Sr, Cs) will be studied. Samples of materials after mutual interaction will be used for this study, in comparison with reference materials.</p>			
<b>Objectives for different specific steps/periods (if applicable):</b> <ul style="list-style-type: none"> <li>• <b><u>Task 1.1: Establishing known State-of-the-Art.</u></b> State-of the art will be prepared, including definition of boundary conditions for experimental studies and details of analytical methods and procedures for sample characterization</li> <li>• <b><u>Task 1.2: Characterization &amp; experimental studies.</u></b></li> <li>• Analyses of in-situ experiment samples: samples of bentonite (Ca-Mg Czech bentonite) and cement (OPC), having been aged under Josef gallery conditions in the previous project, will be analysed in order to determine changes in mechanical/rheological/chemical/transport properties due to mutual interaction (in cooperation with CTU)</li> <li>• Laboratory experiments with bentonite and cement materials (OPC, LPC): ageing of the material under defined temperatures within different time periods, followed by analyses of changes in mechanical/rheological/chemical/transport properties due to mutual bentonite/cement interaction (in cooperation with CTU)</li> <li>• Diffusion studies: determination of changes in bentonite/cement diffusivities due to mutual interaction. Following tracers will be used: <math>^3\text{H}</math>, <math>^{36}\text{Cl}</math> in order to determine changes in porosity and available porosity; Sr, Cs will be used to demonstrate changes in safety relevant nuclides migration behaviour due to material degradation.</li> </ul>			
<b>Main expected outcome:</b> <ul style="list-style-type: none"> <li>• <b><u>Task 1.1: Establishing known State-of-the-Art.</u></b> - State of the art, definition of boundary conditions, experimental approaches</li> <li>• <b><u>Task 1.2: Characterization &amp; experimental studies.</u></b> - Study of changes of bentonite and cement mechanical/rheological/chemical/transport properties due to mutual interaction under in-situ and laboratory conditions (CEC, mineral composition, porosity, permeability, volume density, swelling strength, extent of alkaline</li> </ul>			

## Appendix to Cebama proposal

WP 1			UJV
front; diffusivity) - Influence of mutual interaction on safety function of both materials under repository condition			
<b>Description of work to be performed:</b>  <b>UJV will perform following activities within the project</b> <ul style="list-style-type: none"> <li>• <b><u>Task 1.1: Establishing known State-of-the-Art.</u></b> <ul style="list-style-type: none"> <li>- State of the art, definition of conditions for “ageing procedure”, materials selection (in cooperation with CTU)</li> </ul> </li> <li>• <b><u>Task 1.2: Characterization &amp; experimental studies</u></b> <ul style="list-style-type: none"> <li>- Analyses of in-situ samples (bentonite, OPC) after long term contact (up to 60 month): XRD (mineralogical analyses), CEC and cation availability on exchangeable sites</li> <li>- Analyses of reference samples of materials used (bentonite, OPC, LPC): XRD (mineralogical analyses), CEC and cation availability on exchangeable sites</li> <li>- Analyses of laboratory samples (bentonite, OPC, LPC) after ageing procedure (9/18/27 months): XRD (mineralogical analyses), CEC and cation availability on exchangeable sites</li> <li>- Preparation of aged samples for diffusion studies (bentonite, LPC, OPC)</li> <li>- Diffusion study on bentonite/cement (OPC, LPC) material property changes after mutual interaction: <math>^3\text{H}</math>, <math>^{36}\text{Cl}</math> as conservative tracers will be used; Sr and Cs will be further used in order to determine changes in safety relevant tracer migration in degraded materials</li> </ul> </li> </ul>			
<b>Cooperations</b> <ul style="list-style-type: none"> <li>• UJV Rez will closely cooperate with two CTU faculties: Faculty of Civil Engineering (Centre of Experimental Geotechnics , WP1) and Nuclear Sciences and Physical Engineering (Department of Nuclear Chemistry, WP3). All the institution will be supported by SÚRAO. Each institution operates complementary tools, laboratory equipment and skills. Those all together are needed for successful solution of the complex task-s to be solved within the project.</li> <li>• UJV - CTU FCE: (WP1, inputs to WP3 - CTU providing in-situ and laboratory samples, geotechnical and mechanical tests</li> <li>• UJV - CTU FNSPE - modelling in WP3</li> <li>• UJV, CTU FCE, FNSPE – SÚRAO – all WP</li> <li>• VTT - material exchange (LPC)</li> </ul> The programme is supported by SÚRAO.			
<b>Material exchange with other possible partners is envisaged.</b>			
<b>Added value of Cebama</b> <ul style="list-style-type: none"> <li>• Study of changes of bentonite and cement (OPC, LPC) properties due to mutual interaction under in-situ and laboratory experiments (porosity, CEC, mineral composition, permeability, volume density, swelling pressure, geomechanical properties, diffusivity) under laboratory and in-situ conditions</li> <li>• Influence of mutual interaction on safety function of both materials</li> </ul>			
<b>Planned training activities (if any):</b> Potential for 1 PhD study			
<b>Plans for dissemination:</b>			

## Appendix to Cebama proposal

WP 1			UJV
<ul style="list-style-type: none"><li>• Papers</li><li>• Conference presentations</li></ul>			

## Appendix to Cebama proposal

## 15 / ULOUGH

WP 1			ULOUGH
<b>Overall Objectives:</b> <p>Improved understanding of the changes that occur at mineralogical/geochemical interfaces between cementitious materials and groundwater as a function of key environmental parameters (salinity, pH, redox, carbonation). Established cement formulations relevant to European geological disposal concepts will be addressed. The impact on porosity, permeability and bulk transport properties will be investigated using a variety of methods appropriate to the matrix and the evolving phase assemblage. The experiments will also draw on samples prepared in parallel studies at Uni Sheffield. Results will be used to inform modelling tasks performed by CEBAMA partners in WP3.</p>			
<b>Objectives for different specific steps/periods (if applicable):</b> <ul style="list-style-type: none"> <li>Phase 1 (6 months) – collaboration with partner laboratory to finalise programme, curing of target cements at ambient temperatures, variable gas pressure and water saturation;</li> <li>Phase 2 (36 months) – experiments using synthetic groundwaters to investigate alteration processes and products at cement-(simulated ground)water interfaces. Mineralogical and textural characterisation of samples;</li> <li>Phase 3 (6 months) – synthesis, provision of data for WP3 and final reporting.</li> </ul>			
<b>Main expected outcome:</b> <p>Enhanced understanding of the processes governing interfacial reactions in selected cement formulations in contact with groundwaters. The cements will comprise a low strength, high pH backfill (NRVB - UK) and two low pH cements (silica fume - Finland &amp; Sweden and high alumina cement, HAC - France). The groundwaters will be chosen to be representative of the crystalline Borrowdale Volcanic Group (BVG) and Corallian-Oxfordian Clay (COx). The effect of evolving mineralogy on porosity, (water and gas) permeability and potential bulk transport properties will be investigated by techniques including gas porosimetry, <sup>14</sup>C-PMMA/autoradiography, porewater squeezing and X-rayCT. Derivation of parameters for mechanistic speciation-solubility and chemical transport modelling will allow re-evaluation of simplifying assumptions in safety assessment calculations.</p>			
<b>Description of work to be performed:</b> <ul style="list-style-type: none"> <li>Preparation of cement samples in solutions at two ionic strengths, approximating fresh groundwater and seawater;</li> <li>Periodic solution analysis by ICP-MS, ICP-OES and ion chromatography to obtain compositional data for modelling purposes;</li> <li>Characterisation of solids by (μ)XRD, (μ)XRF, ESEM, TEM;</li> <li>Investigation of the impact of different curing regimes (salinity, controlled carbonation) on evolving mineralogy at the cement-groundwater interfaces;</li> <li>Provision of data to assist thermodynamic modelling by partners under WP3.</li> </ul>			
<b>Cooperation:</b> <p>Uni Loughborough will cooperate closely with its partner laboratory Uni Sheffield. Advisory support will be provided by RWM and the British Geological Survey. Cement formulations and experimental protocols will be based on previous UK and CEC studies. Samples will be shared with</p>			

### Appendix to Cebama proposal

WP 1			ULOUGH
<p>Uni Sheffield for characterisation of physical properties and each partner will be given access to the other's facilities for joint work.</p> <p>The study will draw on extensive experience with cementitious materials at Uni Loughborough across the Chemistry, Materials and Civil Engineering departments. Access will be sought to state of the art phase characterisation facilities at Diamond.</p>			
<p><b>Added value of Cebama:</b> Cebama will benefit from experience gained in national (e.g. NDA) and international (e.g. CEC SKIN) projects.</p>			
<p><b>Planned training activities (if any):</b> Uni Loughborough is a partner in the CEC CINCHII programme developing a pan-European Masters course in Radiochemistry. The PhD student will contribute to this programme.</p>			
<p><b>Plans for dissemination:</b></p> <ul style="list-style-type: none"> <li>• Publication in peer-reviewed journals</li> <li>• Presentations at international (e.g. Migration), IGD-TP and national (NDA) conferences.</li> </ul>			

## Appendix to Cebama proposal

## 16 / CTU

WP 1			CTU
<b>Overall Objectives:</b> <p>The objective of the Czech partners (The CTU – CEG and UJV Řež a.s.) within WP1 is an experimental study on effects of mutual interaction between cement based materials (OPC, LPH) and Czech Ca-Mg bentonite. CTU will focus on mechanical and hydro-physical properties (esp. strength prop., swelling ability, hydraulic conductivity). The works will include laboratory testing of “new” samples after 9 to 27 months of cement-bentonite interaction under defined conditions (high temperature,...). “Old” samples available from previous experiment (testing “cartridges” at Josef URL) will also be tested (aged since 2010). Verification of non-standard methods (strength tests on thin samples) will precede the testing phase. CTU will provide identical samples for the UJV Řež test programme and thus complete data set will be formed. The programme is supported by SÚRAO as the end-user.</p>			
<b>Objectives for different tasks (specific to your organization):</b> <ul style="list-style-type: none"> <li>• <b><u>Task 1.1: Establishing known State-of-the-Art, defining boundary conditions for experimental studies and details of analytical methods for sample characterization</u></b> State of the art, conditions for lab studies</li> <li>• <b><u>Task 1.2: Characterization &amp; experimental studies.</u></b> Testing programme preparation - material, vessels and methods verification, running of “ageing procedures”, sampling and lab testing,</li> </ul>			
<b>Main expected outcome per task (corresponding to list above):</b> <ul style="list-style-type: none"> <li>• <b><u>Task 1.1:</u></b> Test conditions defined</li> <li>• <b><u>Task 1.2:</u></b> Study on effect of long term interaction between cement based materials and bentonite - influence on both bentonite and cement behavior (hydro physical and mechanical prop.)</li> </ul>			
<b>Description of work to be performed (corresponding to list above):</b> <u>Task 1.1:</u> State of the art, definition of conditions for “ageing procedure” <u>Task 1.2:</u> (T1.2.2) Verification of non-standard strength tests methods to be used on thin samples (esp. correlation relationships), materials selection; samples and test vessels preparation, running of “ageing procedures”, sampling (after 9/18/27 months), lab tests on samples (also on samples of “cartridges”, aged since 2010), testing of both – bentonite (hydro-physical prop.) and cement materials (strength tests), providing samples to UJV Řež <ul style="list-style-type: none"> <li>• <b><u>Task 1.3:</u></b> Evaluation of gained data, inputs to WP3 (in cooperation with UJV Řež and potentially other CEBAMA partners)</li> </ul>			
<b>Cooperations:</b> <ul style="list-style-type: none"> <li>• CTU - CEG will closely cooperate with UJV Řež within WP1 on experimental program and analyses and together will provide inputs to WP3. CTU - CEG will take care of samples preparation, mechanical tests on cement samples, bentonite (hydrophysical) tests; UJV will be involved in analyses in bentonite geochemistry and cement analyses. The program is supported by SÚRAO.</li> <li>• Material exchange with other CEBAMA partners is envisaged (e.g. VTT - The Low pH cement).</li> </ul>			

### Appendix to Cebama proposal

WP 1			CTU
<b>Added value of Cebama:</b> <ul style="list-style-type: none"> <li>• Study of changes of bentonite and cement properties due to mutual interaction under in-situ and laboratory experiments (porosity, permeability, volume density, swelling pressure, fluid composition, extent of alkaline front; geomechanical properties);</li> <li>• Influence of mutual interaction on safety function of both materials</li> </ul>			
<b>Planned training activities (if any):</b> Involvement of PhD students (students of CTU in Prague); organizing of CEBAMA PhD workshop at Josef URC			
<b>Plans for dissemination:</b> <ul style="list-style-type: none"> <li>• International conferences, articles in journals, poster in Josef (CTU and SURAO visitors), annual reports of CTU - CEG CTU, leaflet; including of information into the CTU courses focused on RADWASTE disposal</li> </ul>			

## Appendix to Cebama proposal

## 17 / USFD

WP 1			USFD
<b>Overall Objectives:</b> USFD will undertake a detailed physico-chemical characterisation of selected cements relevant to international GDF concepts, including those in a UK context, at the cement / ground water interface. We aim to build a mechanistic understanding of how such interactions are likely influence diffusive transport by advancing understanding of their influence on porosity, permeability and cement mineral phase assemblages as a function of carbonation, pH, salinity and ground water composition. These experiments will be conducted in parallel with ULough, who will focus primarily on the chemical characterisation of these interactions, while USFD will focus on the physical characterisation. High-resolution datasets generated through these experiments will be used to inform modelling tasks performed by CEBAMA partners in WP3.			
<b>Objectives for different tasks (specific to your organization):</b>  <b>Task 1.1: Establishing known State-of-the-Art, defining boundary conditions for experimental studies and details of analytical methods for sample characterization – 3 person months</b> The USFD PhD student will synthesise the first published literature survey on cement materials used in UK GDF concepts, in the context of European GDF concepts.  <b>Task 1.2: Characterization &amp; experimental studies – 30 person months</b> <i>Phase 1 (months 0 - 6):</i> USFD will prepare cements for experimental studies by USFD and ULough. <i>Phase 2 (months 7 – 30):</i> Treatment of samples in groundwater in collaboration with ULough. Determination at USFD of the physico-chemical changes, including those that affect diffusive transport, induced by the groundwater / cement interfacial reactions, linked with ULough chemical characterisation of corresponding samples.			
<b>Main expected outcome per task (corresponding to list above):</b>  <u>Task 1.1:</u> Publishable literature survey pertaining to current state-of-the-art experimental studies on UK GDF cement interfaces in the context of European GDF concepts, for inclusion in a wider WP1 report. <u>Task 1.2:</u> A mechanistic understanding of the processes governing interfacial groundwater / cement reactions under a variety of relevant conditions. Influence of such reactions on the physical properties of cement will be the main focus, and information on cement mineralogical changes will be determined in collaboration with ULough. <u>Task 1.3:</u> A quantitative understanding of how interfacial cement alteration affects microstructure under a variety of relevant groundwater conditions will be determined, including an interpretation of changes to diffusive transport. The latter will be used to inform modelling performed in WP3.			
<b>Description of work to be performed (corresponding to list above):</b>  <u>Task 1.1:</u> With support from RWM (Radioactive Waste Management Ltd.), the USFD PhD student will undertake a wide survey of the literature pertaining to the current state-of-the-art experimental studies on the Nirex Reference Vault Backfill (NRVB) cement, for potential use in a UK intermediate level waste repository in a hard rock scenario. With support from Corkhill, Provis and Hyatt, this review will be synthesised into a publishable report, for addition to the wider CEBAMA Task 1.1. report.  <u>Task 1.2:</u> <u>Phase 1:</u> Cement formulations representative of: (1) low strength, high-pH cement (NRVB); (2) low-			



## Appendix to Cebama proposal

WP 1		USFD
<p>pH, PC - silica fume cement (representative of Swedish and Finnish concepts); and (3) low pH, PC - silica fume - FA blended cement (representative of the French concept) will be prepared at USFD. A subset of cements will be cured in controlled CO<sub>2</sub> conditions, while all other cements will be cured at ambient conditions. Three curing times (short, medium and long-term) will be utilised. Cement samples will be immersed in three ground water solutions, carefully constrained by ULough, representative of: (1) crystalline hard rock; (2) Corallian-Oxfordian Clay (COx); and (3) a high ionic strength solution representative of sea water.</p>		
<p><u>Phase 2:</u> Physical characterisation using porosimetry and permeability techniques (MIP, gas adsorption, oxygen permeametry) and <math>\mu</math>-XCT will be systematically applied to identify changes in porosity, permeability and tortuosity as a function of interfacial alteration by groundwater (e.g. through precipitation / dissolution processes). We will apply for neutron radiography and tomography access at specialist facilities in the UK (e.g. Diamond, ISIS), and collaborate with HZDR to attain high-resolution microstructural datasets of selected altered cements. Changes in cement microstructure will also be monitored using microscopy techniques (SEM, TEM). We will support parallel chemical analysis performed at ULough, by providing expertise in characterisation of secondary cement phases through e.g. NMR and <math>\mu</math>-XAS techniques.</p>		
<p><u>Task 1.3:</u> The detailed datasets generated in Task 1.2. will be combined and investigated through application of diffusive transport modelling. Models will be created using the microstructural and physical observations to describe how the different geochemical conditions (groundwater composition, pH, salinity), curing conditions (ambient, CO<sub>2</sub>-controlled) and curing times are likely to affect transport through the cement material. The USFD student will visit a CEBAMA WP3 partner to maximise the outcomes of this task.</p>		
<p><b>Cooperations:</b> USFD will closely co-operate with ULough in the experimental work planned in Task 1.2, sharing samples, interfacial reaction experiments, expertise in characterisation and providing shared access to laboratory facilities. We hope to collaborate with HZDR in order to apply the GeoPET technique to altered cements. Advisory support from the UK waste management organization, RWM, will be provided, especially for Task 1.1. We hope to collaborate with an appropriate WP3 partner in Task 1.3. (t.b.c.).</p>		
<p><b>Added value of Cebama:</b> This project investigates a unique combination of interfaces which are relevant both to the UK ILW geological disposal scenario and also of interest to other European geo-disposal concepts. State-of-the-art physical characterisation techniques will be applied in conjunction with standard methodologies to support transport modelling.</p>		
<p><b>Planned training activities (if any):</b> The student will benefit from associate membership to the EPSRC Next Generation Nuclear Doctoral Training Centre, including selected week-long MSc level modules in the nuclear fuel cycle. USFD will organize a summer school on public engagement in nuclear sciences, planned for summer 2015; attendance could be opened to young scientists on the CEBAMA project for a small registration fee; the event will be in workshop format with a focus on developing practical skills.</p>		
<p><b>Plans for dissemination:</b> Research outcomes will be published in leading peer-reviewed journals and other high-impact outlets. These will be published with Gold open access where possible, and Green open access via the White Rose repository otherwise. We will present interim results and research outcomes at prominent national (e.g. IoM<sup>3</sup> Cement and Concrete science, RWM PhD conference day) and international conferences (e.g. MRS or Goldschmidt). The project will also be networked with national R&amp;D programs (e.g. EPSRC DISTINCTIVE consortium), providing a dissemination route to the UK academic and user community.</p>		

## Appendix to Cebama proposal

18 / VTT

WP 1			VTT
<p><b>Overall Objectives:</b></p> <p>VTT will develop justified, transparent and credible conceptual model of alkaline plume caused by cementitious material degradation in different groundwater/clay environment. Model will be based on experimental data and theoretical modelling. This will give solid evidence and thus enables the estimation of bentonite stability as well as engineered barrier system safety. The combined effects of pozzolanic materials, leaching volumes and low-pH concrete/ groundwater/ bentonite-clay compositions will be studied in detail. Chemical and physical evolution of changing hydration products and leached compositions will be explored.</p>			
<p><b>Objectives for different tasks (<i>specific to your organization</i>):</b></p> <p>The key questions, addressing the Nordic (KBS-3V) repository conditions, to be answered in this WP regarding OPC-groundwater reactions are: 1) how much hydroxyl –ions are leached from hardened OPC-based structures before the pH decreases below 10, and 2) does the lowered pH withstand potential changes in the environment without restoring to the most stable pH of 10,5? Only after understanding and modeling these questions can the long-term safety and stability of the buffer and backfill bentonite be ensured. The first step is to establish the parameters (Task 1.1) prior to the experimental studies (Task 1.2).</p> <p>This experimental challenge of Task 1.2 has two aspects: kinetic and equilibrium. In kinetics, at least three distinct methods that lower the pH of OPC leachate can be identified:</p> <ul style="list-style-type: none"> <li>• Pozzolanic materials reaction with calcium hydroxide and CSH,</li> <li>• alkalis and calcium leaching due groundwater interaction and</li> <li>• charge balance between OPC leachate and groundwater/bentonite ions.</li> </ul> <p>In equilibrium, the pH is dependent on CSH- and groundwater- compositions.</p> <p>WP3's goal is to create well-justified and credible conceptual models to estimate OPC effect on various groundwater compositions and evaluate potential hydroxyl-ion contents through the repository life-time according mass balance calculations. The experimental studies (Task 1.2) will have two distinct objectives: first defining the kinetics of CSH transformation according to the three above mentioned methods; secondly defining equilibrium conditions of various CSH compositions in various leaching groundwater compositions. Based on the results of Task 1.2, descriptions of the individual and combined mechanism will be presented.</p> <p>Research significance has been identified in bentonite –based geological repositories<sup>1</sup> but the groundwater also serves as an intermediary phase in concrete –crystalline rock, -bentonite, -boom -clay, -OPA clay, -COX clay interactions. In each case, OPC – groundwater interactions offer bases to determine long-term (1,000,000 year) stability and interactions in groundwater leaching environment. Previous studies have been made studying stability of phases simultaneously. In simultaneous studies, previous results have shown precipitation of intermediate phases, fracture network clogging or other local effects. These results are applicable in a certain timespan, but do not offer universal and transparent method to predict the long-term stability and performance of the EBS.</p>			

<sup>1</sup> Koskinen Kari, *Effects of cementitious leachates on the EBS, Posiva 2013-04, (2014)*

## Appendix to Cebama proposal

WP 1			VTT
<p><b>Main expected outcome per task (corresponding to list above):</b></p> <ul style="list-style-type: none"> <li>• <u>Task 1.1:</u> Summary of existing State-of-the art (report) detailing: <ul style="list-style-type: none"> <li>- equilibrium compositions of hydration products in aqueous solutions.</li> <li>- C-S-H/ion interactions.</li> <li>- boundary conditions needed for Task 1.2 and Task 1.3 work (linked to WP3 models).</li> </ul> </li> <li>• <u>Task 1.2:</u> Experimental results on alkaline plume and solids composition development as function of various leaching volumes and low-pH concrete/groundwater/bentonite -compositions.</li> </ul>			
<p><b>Description of work to be performed (corresponding to list above):</b></p> <ul style="list-style-type: none"> <li>• <u>Task 1.1:</u> Literature research will be performed on equilibrium compositions of calcium-silicate-hydrate gels in aqueous solutions. Further on, the research will be expanded to Portland cement/pozzolanic materials and ionic compositions of groundwater and clay (attention towards bentonite). Literature review/summary will be focused on calcium-silicate-hydrate /ion interactions. Various ions can replace silicate ions in C-S-H structure. Also calcium and water molecules can be replaced. Ions also have an effect on C-S-H physical and chemical properties. The goal of the study is to create comprehensive understanding of these effects based on latest literature and past studies/experience. Besides defining the local representative repository groundwater solution and OPC material compositions, nuclear waste management organisations and nuclear regulatory authorities will contribute information about the requirements and restrictions for the experiments. In Finland, close cooperation with Posiva Oy and SKB on this issue (for high-level waste storage). Possible contribution also from STUK (regulatory authority), Fortum and TVO (low- and intermediate-waste storage).</li> <li>• <u>Task 1.2:</u> This experimental task consist of two lines: <ul style="list-style-type: none"> <li>• Kinetic studies of low-pH concrete/ groundwater/ clay (bentonite focus) system with various leaching volumes and solid compositions. This defines the rates of pozzolanic reaction, leaching effect and groundwater interaction compared to each other in incongruent dissolution. Kinetic study enables mass balance calculations for individual effects and gives quantitative information of leaching hydroxyl ions in various scenarios.</li> <li>• Equilibrium studies of low-pH concrete/ groundwater/ clay -compositions. The goal of the study is to determine congruent dissolution composition of various binders, CSH and leachate compositions in various leaching groundwater. Equilibrium study will be performed for selected material and groundwater compositions.</li> </ul> Interaction between cementitious materials and clay/bentonite will be through the groundwater chemistry. Synthetic groundwater as well as real groundwater from long-term site investigations will be used in these investigations.</li> </ul>			

## Appendix to Cebama proposal

WP 1			VTT
<p>Cementitious materials used will be both binary (OPC-Silica) and ternary systems (OPC-Silica- fly ash). A wide range of C/S ratios will be used, i.e. ratio will be from 2-0.5. The water-binder ratios will vary from 0.5 (used for concrete) to 2.0 (towards grouts), to also account for the test protocol in a protective atmosphere. Multiple recipes will be produced or used covering the European dimensions, in exchange with other consortium partners. Batch experiments will be done after aging by water-bentonite storage and then following the common European protocols (clarified in Task 1.1), prior to assessing how the mineralogy is changing due to the leaching and ion-exchange processes. Both powdered/ground and solid samples are used to identify multi-scale implications.</p> <p>The studies include analysis of solid phases and solutions, with the focus on low-pH concretes but also applicable to other material such as shotcrete and rock bolt mortars. Analysis of leached groundwater compositions are performed by using ion selective electrodes, ICP and Si-NMR. Solid compositions, reaction degrees and crystallinity of the phases will be analyzed with XRD, XRF, TGA, Si-NMR, titrations, SEM, SEM-EDX and TEM.</p>			
<p><b>Cooperations:</b></p> <ul style="list-style-type: none"> <li>• <i>Within WP1, comparative studies will be conducted between RTD providers (cooperation expected with CTU/UJV, CSIC, USFD) based on varying material (concrete formulations &amp; clay types) and environmental (groundwater) conditions to gain an array of input parameters. Complimentary studies will be established to optimise a spectrum of results. Pre-existing laboratory and field samples will be exchanged internationally, including materials possessed by WMOs.</i></li> <li>• <i>Close cooperation and iteration with WP3 will be established throughout the work, so that modeling is developed simultaneously. This work package will generate mechanisms and boundary values for modelling and provide quantitative data for independent validation of models.</i></li> <li>• <i>Close cooperation with POSIVA, SKB, ANDRA and other nuclear waste management organisations will be arranged by establishing work package steering group where special technical questions concerning this work package will be addressed.</i></li> </ul>			
<p><b>Added value of Cebama:</b></p> <p>Provide mechanisms and boundary values for cementitious material groundwater interaction modelling. Utilize international existing field samples results for validation of mechanisms, such as aged concrete-bentonite samples immersed in groundwater or repository conditions.</p>			
<p><b>Planned training activities (if any):</b></p> <p>Two PhD-students will use these results in their works (both at Aalto University in Espoo, Finland). VTT has experience in hosting major workshops and events, and thus would be willing to be involved in CEBAMA-related training. Mobility of researchers will be promoted by arranging researcher exchange between partners and potentially between complimentary H2020 or FP7 Euratom projects. .</p>			
<p><b>Plans for dissemination (similar text provided to WP3):</b></p> <ul style="list-style-type: none"> <li>• Two PhD dissertations will use results from the Cebama project in their work. Both works are expected to be finalized during the project duration.</li> <li>• At least 3 scientific journal publications will be generated (i.e. <i>Journal of Cement and Concrete Research</i>, <i>Journal of Applied Materials</i>) along with 3 conference and/or trade</li> </ul>			

**Appendix to Cebama proposal**

WP 1			VTT
<p>journal presentations (such as and IGD-TP events, Posiva Oy and VTT organization's public magazines). VTT will contribute to the Cebama web page and periodic newsletters.</p> <ul style="list-style-type: none"> <li>• VTT as a non-profit research organisations will disseminate the results of this research in the future research and development projects at national and EU level. Through the good connections with their local stakeholders, VTT can spread and promote the technologies developed in this research, thus helping nuclear waste management organisations in their assignment. VTT's own know-how and competence in cement based materials - groundwater - bentonite interaction related subjects will strengthen through this project, and cooperation at EU level will be reinforced.</li> </ul>			

## Appendix to Cebama proposal

## 19 / HZDR

WP1			HZDR
<b>Overall Objectives:</b> HZDR provides the spatio-temporal visualization of transport processes in geological material on the lab scale as 4D data sets for numerical model validation; quantification of changes of effective porosity and permeability by correlated PET/CT interpretation; enhanced process understanding of the interaction between and reaction at interfaces between cementitious and clay formations, by applying the unique non-invasive GeoPET method in joint experiments with SCK and UniBE.			
<b>Objectives for different specific steps/periods (if applicable):</b> <ul style="list-style-type: none"> <li><b><u>Task 1.1 Establishing known State-of-the-Art, defining boundary conditions for experimental studies and details of analytical methods for samples characterization</u></b>              Detailed agreement with Cebama partners SCK and UniBE about the design of experiments, sample preparation, set-up of experiments, scope of the/their modelling approach.</li> <li><b><u>Task 1.2 Characterization &amp; experimental studies</u></b>              Joint experiments with both partners lasting throughout the project phase:             <ul style="list-style-type: none"> <li>4D imaging of pore water transport within and across interfaces between cement based materials and geological formations</li> </ul> </li> </ul> 1.2.1 Interface with claystone: Boom clay in close cooperation with SCK, Opalinus clay in close cooperation with UniBE 1.2.2 Interface with bentonite: MX-80 in close cooperation with UniB <ul style="list-style-type: none"> <li><b><u>Task 1.3 Conceptual modelling and data interpretation</u></b></li> <li>Determination of space-resolved effective transport parameters and their change during leaching/corrosion and/or precipitation/mineral alteration.</li> <li>Final data exchange with partners, documentation and finalizing publications.</li> </ul>			
<b>Main expected outcome:</b> <ul style="list-style-type: none"> <li><b><u>Task 1.1</u></b>              Coordinated design of experiments (sample specimen, applied solutions, experimental boundary conditions)</li> <li><b><u>Task 1.2</u></b>              Quantitative experimental process visualizations of pore water transport within and across interfaces between cement based materials and geological formations.</li> <li><b><u>Task 1.3</u></b>              Quantitative effective transport parameters (<math>D_{eff}</math>, porosity, preferential reaction pathways) and their change in time due to leaching/corrosion and/or precipitation/mineral alteration.</li> <li>Enhanced process understanding in terms of back coupling between reaction and pore fluid transport and the likely development of preferential reaction paths by means of PET/CT overlays.</li> <li>Provision of 4D tracer concentration distributions in samples affected by Ca-leaching and carbonation process (clogging or opening of pores) for up-scaling approach (SCK/BRGM)</li> <li>Provision of 4D tracer concentration distributions in samples affected by corrosion of cement and bentonite at an Opalinus Clay interface (clogging or opening of pores) as benchmark data set for a tools based on OpenGeoSys6-GEM (UniBern/PSI)</li> </ul>			
<b>Description of work to be performed:</b> <b>Task 1.1</b> (Beginning phase 0-6 months) In depth discussion with Cebama partners SCK and UniBern about the design of experiments, reception of sample material from the partners, coordinated set-up of experiments at HZDR in analogy to set-ups at partner institutions, striking an agreement upon type of data to be exchanged/scope of modelling approach.			

## Appendix to Cebama proposal

WP1			HZDR
<p><b>Task 1.2 (Middle phase 7-42 months)</b></p> <p><b>A) Degradation of cement with Boom-clay pore water</b> (Joint Experiment with SCK)</p> <p>Identifying how reacting fluids alter the transport parameters of cementitious components and change the microstructure at the Boom clay-concrete interface (freshly manufactured as well as aged samples) by GeoPET process observation. As reactive fluids Boom clay pore water will be advectively percolated through freshly manufactured cement samples. The cement is a low-strength, high-porosity, high-pH cement mixture representative for the backfill composition in the Belgian disposal concept (NRVB-type). The pore water contains 14 mmol/L of bicarbonate and is expected to cause carbonation and Ca-leaching in the cement. During percolation, the propagation of the penetrating fluids will be visualized by means of PET over time and the pore water composition will be analysed depending on availability. In these experiments, the conditions in the HADES URF can be mimicked (advective flow regime from the clay to the cement).</p> <p>Processes to be quantified: Migration of injected fluids, clogging or opening of transport effective pores in cement component that results in changing flow and transport processes due to porosity and/or permeability changes.</p> <p><b>B) Degradation of aged cement/claystone and cement/bentonite interfaces</b> (with UniBern)</p> <p>Identifying how reacting fluids enter (slow advection and diffusion) the sample specimen and alter the transport parameters with time by GeoPET process observation. Within this cooperation aged cement/Opalinus Clay and cement/MX-80 bentonite interface from an 3-7 years ongoing in-situ experiment at the Mt Terri URL are used. The samples are of relevant scale (~5 cm) and are representative for the Swiss repository concept. The confined apparatus will be sufficiently transparent for X-rays and PET radiation.</p> <p>Processes to be quantified: Migration of injected fluids, clogging or opening of transport effective pores in cement component that results in changing flow and transport processes due to porosity and/or permeability changes.</p> <p><b>Task 1.3 (End phase 33-48 months)</b></p> <ul style="list-style-type: none"> <li>- Determination of effective transport parameters from 4D PET/CT data sets.</li> <li>- Tomographic data and physical and geochemical information on the experiments will be transferred to modellers in WP3 (SCK (Lattice Boltzmann/PHREEQC) and UniBern/PSI: (OGS/GEMS)).</li> </ul>			
<p><b>Cooperations:</b></p> <ul style="list-style-type: none"> <li>• Cooperations with Cebama partners is central to our approach (WP1 (SCK, UniBern) &amp; WP3 (SCK, UniBern/PSI)).</li> <li>• Cebama external partners in respect to CT imaging may be the J. Gutenberg University Mainz, Bundesanstalt für Materialforschung und –prüfung (BAM), and Leipzig University.</li> </ul>			
<p><b>Added value of Cebama:</b></p> <ul style="list-style-type: none"> <li>• Determination of effective transport parameters from 4D PET/CT data sets resulting from well-defined corrosion experiments at cement-clay interfaces.</li> <li>• Enhanced process understanding of corrosion of cement-clay interfaces (post-closure evolution) for supporting the safety case.</li> </ul>			
<p><b>Planned training activities (if any):</b></p> <p><i>Provision of PET/CT images and movies from the transport process observations to partners who conduct training activities.</i></p>			
<p><b>Plans for dissemination:</b></p> <ul style="list-style-type: none"> <li>• Presentation at international conferences, publication of results in peer-reviewed scientific journals</li> </ul>			

## Appendix to Cebama proposal

## 20 / LML

WP 1			LML
<b>Overall Objectives:</b> <p>The LML will develop a series of laboratory tests for the characterization of hydromechanical behavior of the concrete/argillite interface under complex mechanical, hydraulic, hydric, thermal and chemical conditions.</p>			
<b>Objectives for different tasks (<i>specific to your organization</i>):</b> <ul style="list-style-type: none"> <li>• <b><u>Task 1.1: Establishing known State-of-the-Art, defining boundary conditions for experimental studies and details of analytical methods for sample characterization</u></b>  An extensive synthesis will be performed on existing data on the mechanical and hydraulic properties of concrete/argillite interface in order to guide the choice of the sample geometry, the boundary conditions of tests and representative values of temperature, saturation and carbonation process.</li> <li>• <b><u>Task 1.2: Characterization &amp; experimental studies.</u></b>  To determine the mechanical properties and the evolution of permeability of interfaces under normal and shear stress, to characterize the effects of saturation, temperature and carbonation process</li> </ul>			
<b>Main expected outcome per task (corresponding to list above):</b> <ul style="list-style-type: none"> <li>• <b><u>Task 1.1:</u></b> A comprehensive synthesis on the existing data and models on the hydromechanical properties of concrete/argillite interfaces</li> <li>• <b><u>Task 1.2:</u></b> A new data base on the hydromechanical properties of concrete/argillite interfaces</li> </ul>			
<b>Description of work to be performed (corresponding to list above):</b> <ul style="list-style-type: none"> <li>• <b><u>Task 1.1:</u></b> An extensive bibliographic study will be performed. In this study, we will focus on the mechanical properties and permeability of concrete/argillite interfaces. Both existing experimental data and numerical models will be included. A particular attention will be put on the effects of saturation, temperature and carbonation process. Based on this bibliographic study, we will jointly with Andra choose the geometry of sample, boundary conditions for both mechanical and hydromechanical tests, value ranges of temperature, saturation degree; and the carbonation process to be used in Task 1.2.</li> <li>• <b><u>Task 1.2:</u></b> A series of laboratory tests will be performed on the interface between concrete and Cox clay. We will use a low pH mature concrete without artificial aging. The size of sample will be of centimeters. In the previous works, le LML developed an original experimental device allowing the realization of compression/shearing tests on interfaces under different temperature, relative humidity and fluid flow pressure. This device will be improved and</li> </ul>			



## Appendix to Cebama proposal

WP 1			LML
<p>used in the present work. It is proposed to realize four groups of tests.</p> <p>In the group 1, the mechanical behaviour of interface is investigated under room temperature (20°C) and saturated condition (RH=100%), taken as the reference state. It is proposed to perform first normal compression tests with different values of fluid pressure in the interface to characterize the nonlinear deformation (closure) of the interface as a function of normal stress. In particular, we will verify the validity of effective stress concept for the mechanical behaviour of the interface. Also, we will define the critical value of such an effective stress leading to the mechanical “closure” of interface (the normal stiffness becomes close to that of the intact concrete or Cox clay). Then direct shear tests will be conducted under different values of effective normal stress. We will characterize the evolutions of both shear strain (sliding) and normal strain (closure or opening) as functions of shear stress.</p> <p>In the group 2, we propose to study the hydromechanical coupling in the interface under the reference state. The evolution of permeability (or hydraulic conductivity) of the interface will be investigated. It is proposed to perform the measurement of permeability through the interface under different values of normal stress (say 6, 9 and 12MPa) and fluid pressure (say 1, 2 and 4MPa) in the interface.</p> <p>In the group 3, it is proposed to investigate the influences of temperature and relative humidity on the hydromechanical behaviour of interface. For this purpose, a series of normal compression and shear tests, with measurement of permeability, will be performed as described above but under different values of temperature (say 40°C, 60° and 80°C) and relative humidity (say 50%, 70% and 90%).</p> <p>In the group 4, we propose to investigate the effects of chemical degradation on the hydromechanical behaviours of interface. However, the present study will be limited to the carbonation process. Note that very few data exist one the carbonation of low pH concrete. In this task, we try to obtain some preliminary experimental data on the macroscopic characterization of carbonation effects on the hydromechanical properties of interface without wanting to describe in depth the chemical-physical process of carbonation in low pH concrete. For this purpose, the interface will be subjected to an accelerated carbonation process of concrete. For different levels of carbonation characterized by the physical/chemical analysis, normal compression and direct shear tests will be carried out, with the measurement of permeability in order to study the effect of carbonation on the mechanical and hydraulic properties of the interface, in particular on the hydraulic sealing and mechanical compressibility.</p> <p>Based on the experimental data obtained from the laboratory tests conducted in Task 1.2, we will proceed with the conceptual modelling and data interpretation. In particular, the following aspects will be considered.</p> <p>From direct shear tests, we will define the failure criterion of the interface under combined effective normal stress and shear stress, and also the criterion for the occurrence of normal dilatancy (shear induced opening) of the interface.</p> <p>We will establish the relationship between the permeability, effective normal stress and normal strain (interface closure and opening) of the interface.</p> <p>Finally, we will establish the impacts of temperature, relative humidity and carbonation process on the elastic/plastic behaviour, failure criterion and hydromechanical coupling of</p>			

## Appendix to Cebama proposal

WP 1			LML
the interface.			
<b>Cooperations:</b> <ul style="list-style-type: none"> <li><i>The LML will work in a close cooperation with Andra on the choice of sample geometry and testing conditions (boundary conditions, temperature, saturation degree, carbonation process). Also, we will work together on the complementary between laboratory tests (LML) and in situ experiments (Andra) and the size effects on the hydromechanical behaviors of interface.</i></li> <li><i>No cooperation expected with external partners</i></li> </ul>			
<b>Added value of Cebama:</b> Multi-disciplinary and complementary approaches will be a key factor for the success of such a complex project			
<b>Plans for dissemination:</b> <ul style="list-style-type: none"> <li>Publication of papers in peer-reviewed journals</li> <li>Communications in international conferences</li> </ul>			
<b>Deliverables and milestones proposed (the information will be integrated for the respective work packages):</b> A comprehensive state-of-the-art on the hydromechanical behavior of concrete/argillite interfaces An original data base on the hydromechanical behavior of interface and influences of temperature, relative humidity and carbonation Conceptual models and relationships on the mechanical strength and permeability evolution of the interface.			

## Appendix to Cebama proposal

## 21 - UAM

WP 1			UAM
<b>Overall Objectives:</b> <ol style="list-style-type: none"> <li>1. To perform the geochemical study of high pH concrete (OPC)-FEBEX-bentonite and OPC concrete-Grimsel granite interfaces at microscopic scale (<math>\mu\text{m}</math>-mm) within FEBEX in-situ test and HB6 cell long-term experiments</li> <li>2. To determine the characteristic surface interface reactivity (complementary experiments nm-<math>\mu\text{m}</math> scale) of compacted FEBEX bentonite induced by OPC based concrete pore water (high and low pH concrete (OPC and LPH)), resulting from Grimsel water-concrete interaction.</li> </ol>			
<b>Objectives for different tasks (specific to your organization):</b> <ul style="list-style-type: none"> <li>• <b><u>Task 1.1: Establishing known State-of-the-Art, defining boundary conditions for experimental studies and details of analytical methods for sample characterization</u></b> <ol style="list-style-type: none"> <li>1. Setting up of the requirements of sampling and conditioning of in situ large scale at OPC interfaces (FEBEX): (1-6 months)</li> <li>2. Design and implementation of Surface Reactivity Interface Experiments (SERIE) (1-6 months)</li> </ol> </li> <li>• <b><u>Task 1.2: Characterization &amp; experimental studies.</u></b></li> <li>• <b><u>1.2.2. Interfaces with bentonite</u></b> <ol style="list-style-type: none"> <li>1. Characterization of FEBEX interfaces (6-42 month)</li> <li>2. Development, sequential sampling and characterization of SERIE experiments (6-30 month)</li> <li>3. Dismantling of HB6 cell (10 years): sub-sampling and conditioning; characterization (12-38 month)</li> </ol> </li> </ul>			
<b>Main expected outcome per task (corresponding to list above):</b> <ul style="list-style-type: none"> <li>• <b><u>Task 1.1:</u></b> Establishing, according to the previous knowledge, of sub-sampling conditions of both in situ and experimental interfaces. Local interface experimental design: interaction pore waters from BO-ADUS Borehole/ designed OPC and LPH (CSIC incoming) and starting of SERIE experiments.</li> <li>• <b><u>Task 1.2:</u></b> Assessment of the low impact on primary safety functions of EBSs of concrete interface effects, predicted by previous investigations, in terms of mineralogical alteration thickness (nm-<math>\mu\text{m}</math>-mm) from the beginning (nm-pore processes) to long-term HB experiments and in-situ FEBEX (<math>\mu\text{m}</math>-mm).</li> <li>• <b><u>Task 1.3:</u></b> Analyses of different source (time –space scaled) data allowing to set a basis for performance discussion: new-formed by-products at interfaces and how they produce porosity change phenomena affecting transport mostly in OPC-FEBEX interface (LPH comparison at nm scale) Special attention will be focus to secondary phases (e.g. CSH, MSH, Inorganic carbon species) and changes in physical properties that would affect most significantly RN transport.</li> </ul>			
<b>Description of work to be performed (corresponding to list above):</b> <ul style="list-style-type: none"> <li>• <b><u>Task 1.1:</u></b> <ol style="list-style-type: none"> <li>A) Analysis of previous knowledge coming from ECOCLAY(CSI-UAM)-NFPRO(CIEMAT-UAM)-ESRED(CSIC)-PEBS(CIEMAT-UAM) in order to condition selected preserved</li> </ol> </li> </ul>			

## Appendix to Cebama proposal

WP 1			UAM
<p>FEBEX interfaces in cooperation with CIEMAT. They will be cut in the laboratory (already experimented in PEBS avoiding drying) to obtain small specimens including the interfaces. Composite samples will be freeze dried and part of them embedded in low viscosity resin. (1-4 months)</p> <p>B) Design and implementation of <b>SERIE</b> experiments: <b>OPC</b> and <b>LPH / BO-ADUS Borehole</b> interaction porewater (CSIC) will be infiltrated through a small plexiglass cell containing 2-3 mm thickness <b>OPC</b> or <b>LHC</b> mortar/PP filter membrane/2-3 mm of 0.5 mm compacted <b>FEBEX</b> bentonite. The objective is to produce a flat small interface to be analyzed for micro-structural, mineralogical and chemical properties at nm-<math>\mu</math>m scale by means either of easy disassembling of the interface or for direct use for freeze drying and embedment in low viscosity resin. The experiments will last 1 week, 1 month and 6 months (4-12 months). Percolated water will be collected and analyzed for major ions chemistry. At least 2 replica x 2 pore-water x 3 times (12 tests). Temperature of the tests will be set to actual temperature at the in-situ interfaces.</p> <ul style="list-style-type: none"> <li>• <u>Task 1.2:</u> <p>A) Geochemical study of <b>high pH concrete(OPC)-FEBEX</b> interfaces at microscopic scale (<math>\mu</math>m-mm):</p> <p>aa) <b>Samples:</b> FEBEX (<b>OPC-bentonite</b> and OPC-granite interfaces); HB6 cell (existing 10 year <b>OPC-bentonite</b> cell under thermal gradient conditions). All samples coming from experiments implemented by CIEMAT and from dismantling of FEBEX in situ experiments. Conditioning was preplanned in task 1.1.2.</p> <p>bb) <b>Analyses:</b> XRD of <math>\mu</math>m-mm depth from interfaces using small amounts (&lt; 100 mg) samples; SEM-EDX-CL for phase characterization, chemical profiling (<math>\mu</math>m scale depth) and porosity-estimation. BET specific surface of mm sliced samples.</p> <p>B) Complementary experiments to characterize surface interface reactivity (<b>SERIE</b>)</p> <p>aa) <b>Analysis:</b> Mostly the same as task 1.2 bb) with the additional capability for chemical analysis of the percolating effluents and XRD-thin film techniques (GI-XRD) to characterize nano-scale surface deposits in free interfaces. Porosity assessment by this technique should be checked.</p> </li> </ul>			
<p><b>Cooperations:</b></p> <ul style="list-style-type: none"> <li>• <b>CIEMAT/UAM/CSIC (within objective 1):</b> Selection of the more suitable cores of the large-scale test will be first performed and driven by CIEMAT, UAM and CSIC; further the CIEMAT group will provide both in situ samples (from FEBEX and cell HB6 experiment). The CSIC group will hold the task of the characterization of aging of concrete cores for profile microstructural changes, from host rock to bentonite, of concrete components (cement paste and aggregate). CIEMAT-AEG will trace geochemical and petro physical data at a larger scale, and UAM the characterization of interfaces and mineralogical alteration. The integration of CIEMAT-AEG/ UAM /CSIC will try to answer how does chemical interactions affect transport properties at several interfaces of interest in CEBAMA.</li> <li>• <b>CIEMAT-AEG/UAM/CSIC (within objective 2):</b> CSIC will design the OPC concrete used in FEBEX experiment. He will also characterize the concrete components alteration near or at the interface focusing to the ageing of concrete after the infiltration test with granitic ground water (from BO-ADUS Borehole) and effect in different transport properties including porosity. Besides CSIC will design the ad-hoc low-pH (LPH) type of concrete in function of the environmental scenario, for studding pore granitic ground water in the</li> </ul>			

## Appendix to Cebama proposal

WP 1			UAM
<p>aging and transport properties loss of concrete. Different concrete samples and initial concrete leached waters will be distributed to CIEMAT-AEG and UAM for interface experiments.</p> <ul style="list-style-type: none"> <li>• <b>UAM/UniBe/BRGM?</b> • UAM/UniBe/BRGM?. These partners are carrying out nm-mm scale micro-structural-microscopy-XRD based studies involving the physical description of how chemical reactivity affects the porespace in different materials. The cooperation will consist in sharing experience in conditioning, preparation and methodologies for the implementation and the analytical interpretation of preserved or studied interfaces.</li> <li>• <b>CIEMAT-AEG/UAM/CSIC</b> will collaborate with UDC to provide input for modeling from the HB laboratory experiments</li> <li>• <b>Cebama external partners</b></li> <li>• <b>ENRESA:</b> will be interested in founding part of PhD training of laboratory concrete experiments in contact with granitic waters affecting bentonite, including short-term infiltration tests and The up-scaling to long term HB cell experiments implemented and hosted by CIEMAT.</li> </ul>			
<p><b>Added value of Cebama:</b></p> <p>The added value for CEBAMA is to contribute to integrate chemical interactions at micro-scale with physical properties, studied in quasi real scenarios. Collaboration with expert groups in applying reactive transport codes to field experiments.</p>			
<p><b>Planned training activities (if any):</b></p> <p>UAM planned the production of a doctoral thesis shared with CSIC in cooperation with CIEMAT, supported partially in the HB experiments hosted by CIEMAT, considering the development of sustainable fresh alkaline (<b>OPC</b>) and low-pH concretes (<b>LPH</b>) evaluating their performance in contact with granitic waters and bentonite by studying the environmental effects on concrete aging (chemical alteration, dimensional stability, etc.) and the consequences on physical properties to maximize safety after closure, alteration of interface concrete/bentonite and evaluation of transport properties.</p>			
<p><b>Plans for dissemination:</b></p> <p>Participation in technical (within the project) and topic related meetings (i.e., ANDRA clay meeting, NUCEM) or scientific workshops, and subsequent publication. We plan to write several publications including a revision of the previous work in the concrete-clay interface topic will be performed.</p>			

## Appendix to Cebama proposal

## 22 / CSIC

WP 1			CSIC
<b>Overall Objectives:</b> <i>CSIC will contribute to the next objectives:</i> <ol style="list-style-type: none"> <li>1. Evaluation of the aging of high-pH concretes (OPC) alteration from in-situ large-scale long-term experiments (FEBEX in-situ experiment and long term HB6 cell) in contact with different EBS (Concrete/FEBEX bentonite and Concrete/Grimsel granite interfaces) to identify the influence on transport properties due to porosity concrete alterations.</li> <li>2. Assessment of the impact of granitic ground water on transport properties of concrete (high and low-pH) from chemical modifications of solid phases to physical alteration properties affecting porosity.</li> </ol>			
<b>Objectives for different tasks (specific to your organization):</b> <ul style="list-style-type: none"> <li>• <b><u>Task 1.1: Establishing known State-of-the-Art, defining boundary conditions for experimental studies and details of analytical methods for sample characterization</u></b> <ol style="list-style-type: none"> <li>1) Setting up of the requirements of sampling and conditioning of in-situ long term experiments (<i>Time 1-6</i>)</li> <li>2) Implementation of the testing procedures for identification of aging of concrete from in-situ long-term (high pH) and laboratory short-term experiments (high and low pH) with respect alteration of concrete transport properties due to the interaction with EBS (<i>Time 1-6</i>)</li> </ol> <i>Time: 1-6M</i>  <i>Effort: 2MM</i> </li> <li>• <b><u>Task 1.2: Characterization &amp; experimental studies.</u></b> <ol style="list-style-type: none"> <li>1) Evaluation of aging alteration of high-pH concretes in the region between different EBS interfaces from in-situ large-scale long-term experiments. (<i>Time: 6-42</i>)</li> <li>2) Assessment of the impact of granitic ground-waters on transport properties of laboratory high-pH (similar to FEBEX) and low-pH concretes. (<i>Time: 6-42</i>)</li> </ol> <i>Time: 6-36M</i>  <i>Effort: 19MM</i> </li> </ul>			
<b>Main expected outcome per task (corresponding to list above):</b> <ul style="list-style-type: none"> <li>• <b><u>Task 1.1:</u></b> Definition of the main parameters or concrete properties that are affected by ground water attack and definition of the more accurate testing analysis to evaluate those specified parameters. Establishing, according to the previous knowledge, of sampling conditions and characterization for large scale and laboratory scale experiments.</li> <li>• <b><u>Task 1.2:</u></b> <ol style="list-style-type: none"> <li>1) Impact on primary safety functions due to the alteration of the high-pH concrete transport properties within variable interfaces of EBS, involving: <b>Grimsel-granite host-rock/concrete/FEBEX-bentonite and HB6 Cell</b>, in terms of hydrates alteration (C-S-H gel dissolution processes and chemical or mineralogical modifications of cement paste including aggregate interfaces), affecting on porosity and permeability of the concrete matrix and components' interfaces.</li> </ol> </li> </ul>			

## Appendix to Cebama proposal

WP 1			CSIC
2)			<p>Definition and up-scaling of the impact of <b>granitic ground water (from BO-ADUS Borehole)</b> in the physic-chemical and transport properties of <b>high-pH and low-pH concretes</b>, by describing the concrete matrix modifications (new-formed by-products, hydrates dissolution rate, modification of the hydrates composition) promoted at different interface types and levels and the modifications promoted in the waters composition (pH, ions concentration) due to their interaction with concretes. Assessment of the role of these modifications in the porosity change phenomena that accordingly influence the transport parameters.</p>
<p><b>Description of work to be performed (corresponding to list above):</b></p> <ul style="list-style-type: none"> <li>• <b>Task 1.1:</b> Short literature review considering the existing studies involving concrete resistance against ground water aggression in order to define the main parameters influenced that better would explain the degradation suffered by the concrete. Integration of the published results with the self-know-how in order to define the most accurate methodology to evaluate the concrete aging alteration promoted by ground water attack, considering both existing in-situ long-term tests and short-term laboratory tests.</li> <li>• <b>Task 1.2:</b> <ol style="list-style-type: none"> <li>1) <b>Evaluation of aging alteration of high-pH concretes from in-situ large-scale long-term experiments in contact with different EBS.</b> (Start: M 6. End: M 46) <ul style="list-style-type: none"> <li>• Cores from <b>large-scale experiment</b> considering different EBS interfaces in contact with high-pH concrete and also bulk of concrete matrix will be tested in order to identify the <b>different levels of aging of concrete in relation to the nearest EBS</b>. Two cases will be studied: <b>FEDEX</b> (Grimsel granite/high-pH concrete/bentonite interfaces, hydration with Grimsel granitic water, BO-ADUS Borehole), and <b>HB6</b> cell (High-pH Concrete/bentonite interface, hydration with saline water). Conditioning of the available concrete samples and preliminary characterization. Evaluation of the chemical and the physical changes (porosity) evolution of concrete components, mainly cement paste phases and aggregates interfaces that influence the transport properties.</li> <li>• Accurate assessment of the physical-chemical aging profile of concretes due to their interaction with different EBS. Influence of the ground water in the alteration processes.</li> </ul> </li> </ol> <p><u>Methodology:</u></p> <ul style="list-style-type: none"> <li>• <b>Conditioning and physic-chemical analyses:</b> alteration gradients of high-pH concrete from EBS interfaces: porosity (MIP), microcracking variation, volume changes, microstructure alteration (microscopy BSEM-EDX analysis, SEM-EDXs, XRD, DTA-TG, FT-MIR).</li> <li>• <b>Interpretation:</b> Definition of high-pH concrete profile alteration promoted by the different conditions involved in the in-situ long-term experiments considered.</li> </ul> <ol style="list-style-type: none"> <li>2) <b>Assessment of the impact of granitic ground-waters on transport properties of high-pH and low-pH concretes.</b> (Start: M6, End: M 36) <ul style="list-style-type: none"> <li>• <b>Implementation and onset of the infiltration tests</b> with the fabricated concretes, considering granitic waters.</li> <li>• Evaluation of the <b>aging/degradation suffered from high-pH concrete and low-pH concrete</b> in the ground waters conditions considered.</li> </ul> </li> </ol> </li> </ul>			

## Appendix to Cebama proposal

WP 1			CSIC
<p><u>Methodology:</u></p> <ul style="list-style-type: none"> <li>• <b>Design and implementation:</b> OPC CEM-I SR concrete (pH&gt;13), similar to those used in FEBEX experiment, and complementary low-pH concretes (pH&lt;11) based on OPC + plus mineral additions will be designed and fabricated. Both types of concretes will be infiltrated with granite ground water type (from B-O ADUS Borehole site) and the resulting leachates will be analysed. The experiments will last for 24 months and progressive alteration of transport properties of concrete matrixes will be identified through periodic evaluation of the modifications promoted both in the leachates and in the solid phases (including porosity).</li> <li>• <b>Analysis:</b> Mostly the same as Task 1.2, from log-term experiment with regard <b>solid phase alterations</b> thus evaluating the <b>aging of high and low-pH concretes</b> in the aggressive environments, with the additional capability for volume and chemical analysis of the percolating effluents (permeability and porosity).</li> <li>• <b>Interpretation:</b> The experiments will allow the definition of the main processes involved in the aging/modifications occurred in high and low-pH concretes when they are submitted to real granitic ground water attack.</li> <li>• </li> </ul>			
<p><b>Cooperations:</b></p> <p><b>CEBAMA internal Partners</b></p> <ul style="list-style-type: none"> <li>• <b>CIEMAT-AEG/UAM/CSIC (within Task 1.1):</b> Selection of the more suitable cores of the large-scale test will be first performed in collaboration with CIEMAT, UAM and CSIC; further the CIEMAT group will provide both in situ samples (from FEBEX and cell HB6 experiment). The CSIC group will hold the task of the characterization of aging of concrete cores for profile microstructural changes, from granitic host rock to bentonite, of concrete components (cement paste and aggregate). CIEMAT will trace geochemical and petro-physical data at a larger scale, and UAM the characterization of interfaces and mineralogical alteration. The integration of CIEMAT/ UAM /CSIC will try to answer how does chemical interactions affect transport properties at several interfaces of interest in CEBAMA.</li> <li>• <b>CIEMAT-AEG/UAM/CSIC (within Task 1.2):</b> CSIC will design the OPC concrete used in FEBEX experiment. He will also characterize the concrete components alteration near or at the interface focusing on the aging of concrete after the infiltration test with granitic ground water (from BO-ADUS Borehole) and effect in different transport properties including porosity. Besides CSIC will design the ad-hoc low-pH type of concrete in function of the environmental scenario, with the aim of studding the influence of granitic pore water in the aging and transport properties loss. Different concrete samples and initial concrete leached waters will be distributed to CIEMAT and UAM for interface experiments.</li> <li>• <b>VTT (within task 1.2 and 1.3):</b> in the definition of the concretes design by determining criteria/requirements for low-pH concretes implementation in function of the environmental scenario and integration of obtained results.</li> <li>• <b>CIEMAT-AEG/UAM/CSIC</b> will collaborate with UDC to provide input for modeling from the HB laboratory experiment</li> </ul> <p><b>CEBAMA External partners</b></p> <ul style="list-style-type: none"> <li>• <b>ENRESA:</b> will be interested in founding part of PhD training of laboratory concrete experiments in contact with granitic waters affecting bentonite, including short-term</li> </ul>			



## Appendix to Cebama proposal

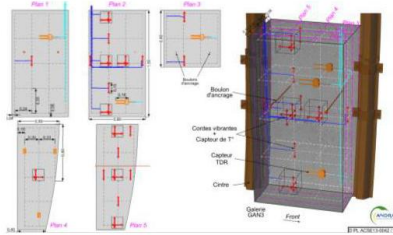
WP 1			CSIC
infiltration tests and The up-scaling to long term HB cell experiments implemented and hosted by CIEMAT.			
<b>Added value of Cebama:</b> Identification of aging processes of high-pH and low-pH concretes in contact with real EBSs. Alteration of physical-chemical properties and consequences in transport phenomena.			
<b>Planned training activities (if any):</b> CSIC planned the production of a doctoral thesis shared with UAM and partially supported in the HB experiments hosted by CIEMAT, considering the development of sustainable alkaline and low-pH concretes, evaluating their performance in contact with granitic waters and bentonite by studying the environmental effects on concrete aging (chemical alteration, dimensional stability, etc.) and the consequences on physical properties to maximize safety after closure, and alteration of interface concrete /bentonite and transport properties			
<b>Plans for dissemination:</b> Participation in topic related meetings or scientific workshops, as NUWCEM, being part of the scientific committees and subsequent publication in recognized construction journals....			

## Appendix to Cebama proposal

## 23 / ANDRA

WP 1			ANDRA
<b>Overall Objectives:</b> <i>Quantification of physical properties evolution (mechanical and transfer) on clay host rock/low pH concrete interfaces</i>			
<b>Objectives for different tasks (specific to your organization):</b> <ul style="list-style-type: none"> <li>• <b><u>Task 1.1: Establishing known State-of-the-Art, design of experiment</u></b> Design and start the experiment in the French URL</li> <li>• <b><u>Task 1.2: Characterization &amp; experimental studies</u></b> Data acquisition and physical behaviour assessment of the structure</li> <li>• <b><u>Task 1.3 Data interpretation</u></b> Model validation of HM behaviour for low pH cements</li> </ul>			
<b>Main expected outcome per task (corresponding to list above):</b> <i>In situ</i> experiment within the French Underground Research Laboratory (URL) at Bure : evolution of low pH concrete walls in contact with Callovo-Oxfordian claystone <ul style="list-style-type: none"> <li>• THM evolution during setting, delayed HM properties evolution with time at Clay/concrete interface</li> <li>• Atmospheric carbonation of low pH concrete in contact with ventilation air (HR variation in carbonated atmosphere)</li> <li>• Mineralogical evolution at concrete/clay host rock interface</li> <li>• Application to concrete wall evolution during the operating period</li> </ul>			
<b>Description of work to be performed (corresponding to list above):</b> <ul style="list-style-type: none"> <li>• <b><u>Task 1.1:</u></b>            Concept designs for nuclear waste repository consider a large use of concrete for structures as well as seals. In the context of clay host rock, low pH concrete is planned to be used in the sealing zone to prevent long term alterations of the swelling clay such as bentonite and the clay host rock. The weakest part of such a system is the interface between the clay and the concrete. Physical and chemical evolution on both sides of this interface will contribute to a major part of the global performance evolution of the seals. Such an evolution has to be understood to report long term performance assessment.             The goal of the proposed experiment is to assess the low pH concrete physical and chemical evolution with time in operating conditions. The two sides of the concrete will be studied: (i) the face in contact with ventilation air which will evolve under atmospheric conditions and will be submit to carbonation (ii) the concrete/clay interface where hydro-mechanical properties will evolve mainly due to geochemical interaction between both materials. Two formulations are considered in this experiment, using ternary blends (CEM I/Silica fume/fly ashes and CEM I/silica fume/blast furnace slag).         </li> <li>• <b><u>Task 1.2:</u></b>            The experiment consists in two thick walls corresponding to two specific low pH formulations. Sensors displayed inside the walls will give a quantitative evolution of the physical behaviour with time (during the setting and during the aerated period). Temperature is monitored and         </li> </ul>			

## Appendix to Cebama proposal

WP 1			ANDRA
<p>vibrating wire sensors as well as optic fibers give the structural evolution (mainly endogenous and drying shrinkage) of the walls. TDR sensors give the water content evolution with time (hydration and drying due to ventilation). In parallel, small samples (concrete cylinder) taken as reference in adiabatic condition, give reference behaviour to calibrate sensors results in the walls.</p>  <p>This experiment will be used as input data to describe the THM behaviour of such specific concrete in <i>in situ</i> conditions for massive elements (thick walls or plugs) in contact with the clay host rock (input data for the WP3).</p>			
<p><b>Cooperations:</b></p> <p><i>This experiment is a field-test to be undertaken in the French URL in Bure.</i></p> <p><i>Outputs from this study are of direct relevance to other WMO programs with regards to seals composition and behaviour expectations</i></p> <p><b>CEBAMA internal partners</b></p> <ul style="list-style-type: none"> <li>• BGS : HM behaviour of sound concrete/clay interface</li> <li>• LML : HM behaviour of degraded concrete/clay interface</li> </ul> <p><b>CEBAMA external partner</b></p> <ul style="list-style-type: none"> <li>• LMDC – INSA Toulouse : THM modelling</li> </ul> <p><i>(LMDC : Laboratoire de Mécanique et Durabilité des Constructions – INSA de Toulouse)</i></p>			
<p><b>Added value of Cebama:</b></p> <p>Description of the physical evolution of the clay/concrete interface</p>			
<p><b>Plans for dissemination:</b></p> <ul style="list-style-type: none"> <li>• Meeting with other partners involved in the project</li> <li>• At least two publications could be submitted on the experiment (design, set up and measurements) and its modelling (early age THM evolution, long term mechanical behaviour)</li> <li>• WP 1 Technical meeting presentations and general assembly</li> </ul>			

## Appendix to Cebama proposal

## 25 / UNIBERN

WP 1			UNIBERN
<b>Overall Objectives:</b> <i>UniBern</i> will perform transport experiments on existing 3-7 year-old interfaces of claystone/concrete and image on-line the time-evolution of the long-term experiments. Both, the stationary phase will be resolved as well as the mobile phase, and a detailed post-mortem characterization will be performed. A complete description of the state and time-evolution of a realistic interface system is gained and possible consequences for radionuclide transport can be directly deduced, and how to consider such interfaces quantitatively in performance assessment.			
<b>Objectives for different tasks (<i>specific to your organization</i>):</b> <ul style="list-style-type: none"> <li><u>Task 1.1: Establishing known State-of-the-Art, defining boundary conditions for experimental studies and details of analytical methods for sample characterization</u> Focus on concrete-claystone interface experiments performed in-situ, and how to transfer boundary conditions into a laboratory set-up.</li> <li><u>Task 1.2.1: Characterization &amp; experimental studies: interfaces with claystone.</u> Set-up and carry out 3 confined transport experiments, one in collaboration with HZDR.</li> </ul>			
<b>Main expected outcome per task (corresponding to list above):</b> <ul style="list-style-type: none"> <li><u>Task 1.1:</u> (a) Identification of geochemical processes and physic-chemical evolution observed in in-situ experiments of concrete-claystone interaction. (b) Experimental boundary conditions for experiments and choice of analytical methods.</li> <li><u>Task 1.2.1:</u> (a) Experiments on concrete/clay interfaces aged over 3-7 years in a realistic environment (e.g. Mont Terri in-situ experiment) will give results on observed transport parameters directly relevant for the situation expected in deep disposal.</li> </ul>			
<b>Description of work to be performed (corresponding to list above):</b> <ul style="list-style-type: none"> <li><u>Task 1.1:</u> Month 0-6 (10%): State-of-the-art review of geochemical processes and physic-chemical evolution observed in in-situ experiments of concrete-claystone interaction, and definition of experimental boundary conditions and best-available techniques for sample and transport process characterisation.</li> <li><u>Task 1.2.1:</u> Month 6-42 (65%): A new generation of X-ray transparent core infiltration device is constructed to fit both high-resolution industrial X-ray CT equipment and PET (positron emission) and SPECT (single-photon-emission) scanners. Reactive transport experiments can be performed on existing 3-7 year-old interface samples (concrete-claystone), recording the chemical condition of the (very slowly) percolating fluid. Time series with tomography techniques image the state and evolution of the stationary phase (X-ray CT) and mobile phase (PET, SPECT), complemented with detailed post-mortem analysis including advanced impregnation techniques (PMMA).</li> </ul>			
<b>Cooperations:</b> <ul style="list-style-type: none"> <li><i>explain your cooperations with Cebama partners in this Workpackage and with</i> <ul style="list-style-type: none"> <li>○ HZDR for PET: joint experiment with equipment from UniBern stationed at HZDR for intermittent PET with long-lived tracer.</li> <li>○ BRGM for <sup>14</sup>C-PMMA impregnation and imaging techniques.</li> </ul> </li> </ul>			

## Appendix to Cebama proposal

WP 1			UNIBERN
<ul style="list-style-type: none"> <li>○ UAM, BRGM, CIEMAT: "open-laboratory policy" for close collaboration regarding sample characterization and complementary methods.</li> <li>• <i>Cebama external partners</i> <ul style="list-style-type: none"> <li>○ CI-Project Consortium, Mont Terri URL (Nagra-Andra- SCK-Obayashi-CRIEPI) to provide existing samples of claystone-concrete (2 types, and support for 2015 drilling campaign for aged interfaces with mortar. Extensive interface characterization was and is performed within the CI Project by multiple scientific teams.</li> <li>○ Uni Fribourg / Geology, for X-ray tomography with new Bruker multiscale nanotomograph SkyScan2211 Nano-CT.</li> <li>○ PSI / Pharmacology Group, for applying single-photon-emission computed tomography (SPECT) if feasibility tests are promising.</li> </ul> </li> </ul>			
<b>Added value of Cebama:</b> (1) Cebama offers excellent platform for joint analytical techniques, tomography in particular, including on-site support; (2) Exchange of state-of-the-art in interface mineralogical/geochemical analysis; (3) Access to advanced reactive transport modelling tools; (4) Platform for discussing data interpretation, process understanding and how this impacts current issues of performance assessment.			
<b>Planned training activities (if any):</b> Basic training for PhD candidate in sample characterization and mineralogical/geochemical analysis, according to needs (Uni Bern); training in X-ray CT technique and equipment operation (at Uni Fribourg); PET technique (at HZDR)			
<b>Plans for dissemination:</b> <ul style="list-style-type: none"> <li>• Presentation at international conferences (scientific and rad-waste specific)</li> <li>• Publication of results in peer-reviewed scientific journals</li> </ul>			

## Appendix to Cebama proposal

## 26 / IRSN

WP 1			IRSN
<b>Overall Objectives:</b> <p>As part of its overall mission of technical support from the Nuclear Safety Authority (ASN), IRSN conducts evaluations on the safety of all operations associated with the management of waste from nuclear industry, and in particular the deep geological disposal of ILL and HL radioactive waste.</p> <p>In order to optimize these evaluations, IRSN develops research actions to identify the physical-chemical evolution of the different concretes foreseen to be used in contact with clays in the DGR.</p>			
<b>Objectives for different tasks (<i>specific to your organization</i>):</b> <ul style="list-style-type: none"> <li>• <b><u>Task 1.1: Establishing known State-of-the-Art, defining boundary conditions for experimental studies and details of analytical methods for sample characterization</u></b> Establish a State-of-the-Art concerning the geochemical evolution of cementitious materials placed in clayey environment between 50°C and 80°C.</li> <li>• <b><u>Task 1.2: Characterization &amp; experimental studies.</u></b> Improve understanding of chemical processes influence at the clay/concrete interfaces on the porosity and transport properties evolutions at 70°C. Focus on new cementitious materials.</li> </ul>			
<b>Main expected outcome per task (corresponding to list above):</b> <ul style="list-style-type: none"> <li>• <b><u>Task 1.1:</u></b> Obtain a synthesis of the state-of-the-Art concerning the evolution of cementitious materials in temperature.</li> <li>• <b><u>Task 1.2:</u></b> Obtain validation data to improve modelling.</li> </ul>			
<b>Description of work to be performed (corresponding to list above):</b> <ul style="list-style-type: none"> <li>• <b><u>Task 1.1:</u></b></li> <li>• <b><u>Task 1.2:</u></b> This work will be performed as part of the PhD of Philippines Lalan (2013-2016). Laboratory tests (through diffusion) of cement pastes/mudstone interfaces: evaluate the influence of chemical processes on the physical-chemical evolution of interfaces and on their transport properties. On-going in situ tests of concretes (Low-pH, CEM I) / argillite in the Tournemire Underground Research Laboratory (CEMTEX project: CEMent Temperature EXperiment) at 70°C: identification of the physical-chemical evolutions at the cementitious/argillites interfaces after 1, 2 and 5 years of interaction. New in situ tests with the new formulations (binary mix) of low-pH concretes envisaged to be used in the French geological waste disposal.</li> </ul>			
<b>Cooperations:</b> <ul style="list-style-type: none"> <li>• <i>Cebama partners : None at the moment, but open to discussion and collaboration</i></li> <li>• <i>Cebama external partners : BEL V, Mines ParisTech</i></li> </ul>			
<b>Added value of Cebama:</b> <p>Our project aims to develop experiments on interfaces to better understand/predict the impact of chemical interactions on the physical properties. The IRSN is specialized for 15 years in</p>			

### Appendix to Cebama proposal

WP 1			IRSN
clay/concrete interactions study. We aim to cross-fertilize our studies with studies carried out by other research teams (large panel of specialist studying the interfaces systems).			
<b>Plans for dissemination:</b> <ul style="list-style-type: none"> <li>• International conferences</li> <li>• Journal papers</li> </ul>			

## APPENDIX 2: Partner Summary Slides



### WP1 Overview – Whole Project

Francis Claret (BRGM), Erika Holt (VTT), Urs Mäder (UniBern)  
 Project Kick-off Meeting (Month 1)  
 Brussels, Belgium  
 2 July 2015



### Project Overview

- 19 of 27 partners participating, averaging 22 Person-months each
- 12 of 13 countries represented (ResOrgs, Universities, WMO)
- WP1 Budget = 2.8 M€ (of 5.9 M€, 47% - near EUG request)
- WP1 Effort = 420.5 Person-months (of 883 PM, 48%)
- **Objective:** Understand how chemical reactions affect porosity, water and gas transport properties at the interface for the 6 following systems. These aspects are investigated by laboratory tests and up-scaling by utilization of in-situ tests (both ongoing and new tests).
  - Low pH & high pH cementitious component – crystalline rock
  - Low pH & high pH cementitious component - bentonite
  - Low pH & high pH cementitious component - OPA, COX, Boom clay





## Project Scope: Experiments

- **Analyses of the interface reactions in contact with different solutions (salinity, pH, redox, carbonates) with respect to changes in mineralogy and porosity evolution on**
  - Low pH cement with bentonite
  - High pH cement (OPC) with Ca-Mg bentonite
- **Characterization of aged cement/mortar/concrete samples in contact with natural host rocks or bentonite with respect to their changes in physical transport parameters, mineralogy and microstructure affecting transport due to mineralogical alteration (e.g. Ca leaching, carbonation) for**
  - High pH concrete / Boom Clay systems
  - High pH cement – Febex bentonite and Grimsel granite
  - High-pH cement – Josef underground laboratory granite
- **Characterization of samples in contact with natural host rocks at 70°C with respect to their changes in physical transport parameters, mineralogy and microstructure affecting transport due to mineralogical alteration (due to Ca leaching and carbonation) for**
  - High pH cement / Tournemire (or Toarcian) / Argillite
  - Low pH cement / Tournemire (or Toarcian) / Argillite

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## Project Scope: Experiments (continued)

- **The mechanical behaviour and the related transport properties of the interface Cement/ Clay, (e.g. OPA, COX).**
  - Quantification of mechanical, hydromechanical behaviour and swelling ability of the low pH concrete/argillite interface under complex mechanical, hydraulic, hydric, thermal and chemical conditions.
  - Derivation of a failure criterion of the interface to be used in Thermo-Hydro-Mechanical-Chemical (THMC) modelling.
- **Diffusion /column experiments to quantify transport parameters including time-lapse experiments (electromigration, periodic tomography).**
  - Low pH and high pH cement systems
  - Concrete / claystone interfaces
- **Methodological work**
  - X-ray computed tomography imaging of the pore structure
  - Complex electrical conductivity measurements on low-pH concrete in contact with pore water
  - Spatio-temporal visualization
- **(Interaction with WP3 inputs and validation)**

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## Project Scope: Material Parameters

Cement material	Claystone	Bentonite	Other rock	Aged interface	Fresh interface	Interface to solution
OPC	SCK CEN	KIT, CIEMAT	CIEMAT	CIEMAT, RWMC, BGS, BRGM, SCK CEN, UJV, HZRD, LML, UAM, CSK, UNIBERN, IRSN	TU Delft, UJV, CTU, UAM, SCK CEN, ANDRA, IRSN	KIT, USFD, ULOUGH, VTT
OPC + LPH	BRGM, ULOUGH, HZDR, UNIBERN, IRSN, TU Delft	UJV, RWMC, HZDR, UAM, CSIC	CSIC, ULOUGH, USFD			
LPH	LML, ANDRA	VTT, BGS	USFD, ULOUGH			

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## Project Scope: Materials & Methods

New Field experiments: Bure (Andra), Tournemire (IRSN), Josef (CZ-UJV & CTU). **Others?**

Existing samples: Bure URL (Andra), Mont Terri URL, Hades URL, Tournemire URL (IRSN), FEBEX @Grimsel (CIEMAT, UAM, CSIC), Japanese URL (RWMC). **Others?**

New Lab sample exchanges: VTT-Andra-CSIC-UJV/CTU. **Others?**

- Diffusion, batch, in-situ: KIT, SCK CEN, UJV, BGS, RWMC, ULOUGH, CTU, USFD, VTT, UAM, ANDRA, IRSN
- Column, advection+diffusion, electromigration: CIEMAT, TU-DELFT, SCK CE, ULOUGH, HZDR, CSIC, UNIBERN
- Geotechnical aspects: LML, BGS, ANDRA, VTT, CTU, RWMC

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## Project Scope: Methods (continued)

Transport with tracers: dissolves gases, stable isotopes, trace components, radionuclide tracers

- Use of tomography (PET), and tracers with ICP-AES, ICP-MS, IC, CRDS, gamma counting, IS
- Characterization with SEM-EDXA, XRD, spectroscopy (EXAFS, XANES, XAS, FTIR, RAMAN), TGA, NMR. Porosity by 2D imaging & MIP, BET,  $\mu$ -CT. Swelling pressure, hydraulic conductivity, pozzolanic reactivity.

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## Project Scope: Milestones

- M1.1 Establish test methods, boundary conditions, materials @ M6
- M1.2 Distribution of first concrete and bentonite samples from existing in-situ (field) sites @ M12
- M1.3 Distribution of first laboratory prepared samples @ M12
- M1.4 Contributing to Kick-off meeting, Annual Project Workshops, Final Workshop, @M1, 12, 24, 36, 48
- M1.5 1<sup>st</sup> WP1 data freeze of experimental data (input to WP3) @ M24
- M1.6 2<sup>nd</sup> WP1 data freeze of experimental data (input to WP3) @ M36

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## Project Scope: Outcomes

- Direct Deliverables:
  - D1.01 Detailed WP1 description of scientific work: compilation of partner descriptions with update of Appendix B (M01 – June 2015)
  - D1.02 Agreement and documentation of systems to be studied (M06 – Nov 2015)
  - D1.03 State-of-art report related to WP1 topics (initial). (M09 – Feb 2016)
  - D1.04 Report on WP1 established experimental boundary conditions, experimental methods. (M10 – March 2016)
  - D1.05 Report on WP1 selected experimental materials to be used, including both new laboratory and aged in-situ samples (M12 – May 2016)
  - D1.06 State-of-art report related to WP1 topics (updated). (M42)
  - D1.07 Manuscript for peer-reviewed publication on results generated in WP1. (M48).
- Indirect Deliverables (all partners):
  - Results Contributed to Annual Workshops (M12, 24, 36, 48) and related proceedings (D4.08, D4.11, D4.13, D4.19)

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## Project Scope: Risks

- Material availability for in-situ experiments or laboratory preparations is delayed in sampling/delivery
- Failure in sample preparation (i.e. delayed reactivity)
- Problems with experimental equipment (i.e. risk with anoxic gloveboxes excluding oxygen & carbonate)
- Delay in obtaining or delivering quantitative results (to WP3 modelling)
- Partners working independently, lacking cooperation
- Lack of adequate PhD students for work implementation
- **OTHERS?**

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## Project Scope: Deviations from Plan

- #10 RWMC: Yearly reporting may deviate based on Japanese national project (calendar year). Some mechanical properties already obtained (available).
- **OTHERS?**

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## Project Scope: Next Step - WP1 Work Meeting

- Scope:
  - Evaluation of test methods & boundary conditions
  - Evaluation of materials under investigation (aged, new casting)
  - Exchange of samples between partners
  - Defining important parameters for integration in WP3
  - **Xxx (other?)**
- Meeting serves as inputs to D1.02, D1.03 and D1.04
- Suggested dates for WP1 specific meeting (duration?):
  - 1<sup>st</sup> Linked with IGD-TP Exchange Forum (London, 3-4.11.15, Month 6)?
  - Then at 1<sup>st</sup> Annual Workshop – May 2016 (Month 12)
  - Maybe linked with Waste-Cement Modeling Workshop in Switzerland (22-25.5.16). [http://empa.ch/plugin/template/empa/\\*/147606](http://empa.ch/plugin/template/empa/*/147606)
  - **Other? (dates, location, etc...)**

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## INDIVIDUAL PARTNER SUMMARIES

*(19 partners x 2 slides each)*

**Speaking 1-2 minutes each.**

Share Highlights only – cooperation interests (methods, materials, input to models)

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### **WP1: # 1 KIT Contribution**

**Prepared by: Marcus Altmaier. Effort: 8 Person-months**

#### **Overall objectives:**

- Experiments on interface reactions of low pH cementitious materials in contact with clay-water-systems.
- Quantification of precipitation/dissolution processes in pores, changes in pore size / distributions and changes in permeability by diffusion experiments.
- Provide sound experimental data for numerical modeling in WP3.

#### **Available Materials/Samples:**

- Design / construction of diffusion cells, set-up of experiments, pumps, filters, sampling
- XRD, SEM for identification of the initial states
- Selection of standard low pH materials according CEM II/A-D or CEM III.
- Samples of low pH concrete materials are available which have been stored under water saturated atmospheric conditions for more than 6 months.
- pH tests confirmed complete hydration.

#### **Any Deviations from Original Project Plan?:**

None





## WP1: #1 KIT, continued

### Description of Work :

#### Task 1.1: Defining boundary conditions for experimental studies (0-12 months)

- Selection of an adequate clay pore water (COX, Boom, OPA) characterization
- Set-up of batch interaction experiments.
- Set-up of samples, diffusion experiments.
- Characterization of solids by SEM methods with respect to their porosity, the mineralogy will be analyzed by XRD and SEM-EDX.
- Determination of initial diffusion properties with radioactive inert tracers (HTO) by breakthrough measurements. Directly after the start of the diffusion experiments with clay pore water.

#### Task 1.2: Characterization & experimental studies (12-24 months)

- Non-equilibrium diffusion experiments in low-pH cement formulations (Cl, I, HTO, Be)
- Repetition of the breakthrough tests using the same samples after different interaction times with the clay pore water in order to determine changes and deviations in the diffusion parameters.
- Quantification of chemical degradation (carbonation of low pH concrete)

#### Solid-liquid interface investigation (12-36 months):

- Analysis of changes in mineralogical composition and porosity of cement surface layers by surface by XRD and SEM-EDX
- Post-mortem analysis of the concrete samples (SEM, XRD, autoradiography), porosity measurements, time-dependent surface investigations of batch samples will be performed after the end of the diffusion experiments (not in the first reporting period).



## WP1: # 3 BRGM Contribution

Prepared by: F. Claret Effort: 17.5 Person-months

### Overall objectives:

- BRGM will perform characterization of Clay/cement materials interfaces. The combined evolution of the mineralogy and microstructure in both materials will be quantify and spatialize in order to implement the data in model to describe the transport properties evolution of the different materials. **This work will be conducted in close collaboration with SCK-CEN** as the studied clay/cement interfaces (either coming from their underground lab or realized in the lab during this project) will be provided by them

### Available Materials/Samples:

- Boom Clay/concrete interfaces samples taken from the HADES underground research facility (URF) (Mol, Belgium). Samples will be taken from the Connecting Gallery, in which low-permeability high-pH concrete wedgeblocks are in contact with Boom Clay for 12 years.
- Percolation type experiments

### Any Deviations from Original Project Plan?:

- No



## WP1: (BRGM), continued

### Description of Work to be Performed/Tasks:

- Task 1.1: Establishing known State-of-the-Art, defining boundary conditions for experimental studies and details of analytical methods for sample characterization
  - Joint effort with SCK-CEN to define the strategy of in-situ concrete/BC interface sampling (0-6 Month)
- Task 1.2: Characterization & experimental studies (jointly with SCK-CEN) (6 to 42 Month)
  - **Multi-scale investigation** to identify any evidence of perturbation in the cement-based materials and the clay.
  - Chemical, mineralogical and  $\mu$ -structural measurements will be performed. Among others, samples selection is on a first screening using computer tomography.
  - The association of several 2D/3D techniques (mineral cartography, autoradiography,  $\mu$ Tomography-RX, SEM, FIB-nT and TEM) allow to reach a quantitative and spatial distribution of the mineralogy and the pore network, from nanometer to micrometer. Therefore, the influence of the mineralogical and chemical changes on the microstructure will be obtained by:
    - a) 2D quantitative spatial distribution of the mineralogy and its evolution with the effect of the perturbation,
    - b) 2D quantitative spatial distribution of the porosity to localize spatial heterogeneities and porosity evolution as regard to the interface,
    - c) focus on localized area to reach 3D pore network
  - Characterization work will be performed in accordance with sample availability
- Task 1.3: Conceptual modeling and data interpretation. (When the data are available)
  - Obtained quantitative data (such as the pore size distribution and the associated mineral distributions) will be used to support lattice Boltzmann simulation to model the diffusion of radionuclides from the pore scale to the pore network

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## WP1: #4 NERC-BGS Contribution

Prepared by: Fiona McEvoy. Effort: 22.5 Person-months

### Overall objectives:

- Understand the interaction/reaction of low-alkali cement leachate on bentonite seal performance with specific emphasis on the impact on strength and permeability of key system interfaces (in particular interaction between seal, host-rock and canister).
- Develop a conceptual model describing seal behavior including identifying and quantifying key processes that occur.
- Reduce uncertainty of long-term seal integrity and performance for inclusion in future predictive numerical models.

### Available Materials/Samples:

- Low-alkali cement (recipe to be provided by ANDRA), COx (provided by ANDRA), bentonite, carbon steel, synthetic porewater and low-alkali fluid (specifications provided by ANDRA)

**Any Deviations from Original Project Plan?: No**

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## WP1: (NERC-BGS), continued

### Description of Work to be Performed/Tasks:

#### Preparatory and preliminary characterisation phase (Year 1-2)

- Design, build and commission experimental apparatus
- Prepare materials (COx, carbon steel, bentonite) and testing permeants
- Mechanical testing of carbon steel, and young and aged low-alkali cement
- Determination of swelling capacity of COx

#### Main experimental phase (Year 2-3)

Undertake experimental programme to measure hydromechanical and transport properties of the following interface properties under normal load and shear for: COx/COx; Low-alkali cement/COx; Low-alkali cement/carbon steel; Low-alkali cement/bentonite; and COx/carbon steel with a fixed interface gap.

#### Post-test characterisation phase (Year 4)

X-ray imaging, optical microscopy and analysis of the pathways highlighted by pore fluid tagging to investigate distribution of flow paths along the interface.

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## WP1# 5 CIEMAT Contribution

Prepared by: María J. Turrero

Effort: 16 Person-months

### Overall objectives:

- Hydro-geo-chemical interactions of high pH concrete and FEBEX bentonite from in situ (FEBEX) and laboratory experiments
- Quantification of porosity change phenomena affecting transport and extent of the alteration of physico-chemical properties of the materials.

### Available Materials/Samples:

- FEBEX bentonite in contact with OPC from the HB6 laboratory experiment lasted 10 years. Scale 1:10. 1-D hydration from the concrete. **Samples shared with UAM and CSIC.** Results will be modelled by UDC.
- FEBEX bentonite in contact with OPC from the in situ FEBEX experiment (GTS). Scale 1:1. Radial hydration from the host rock. **Samples shared with UAM and CSIC.**

### Any Deviations from Original Project Plan?:

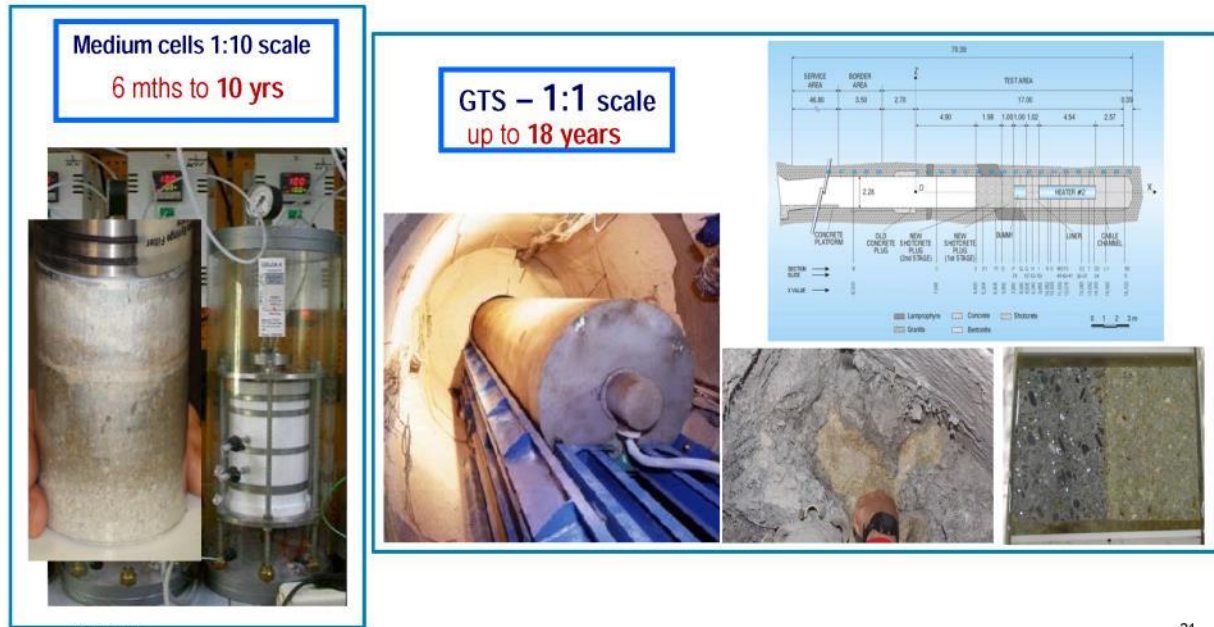
- No

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Prepared by: María J. Turrero  
Effort: 16 Person-months



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## Description of Work to be Performed/Tasks:

- Task 1.1 - State-of-the-Art (month 1 to 6)
  - Conditioning of the samples coming from the in situ experiment.
  - Dismantling, sampling and conditioning of the samples coming from the laboratory experiments.
  - Decisions on characterization procedures of the experiments.
- Task 1.2: Characterization & experimental studies (month 6 to 42)
  - Hydrogeochemical evolution of the EBS at interface and larger scale with emphasis in the alkaline plume: integration of hydro-geochemical results of the experiments plus determination of natural chemical tracers to interpret geochemical processes.
  - Analysis of extractable soluble salts of the EBS materials and bentonite exchangeable cations in order to have indicators of major elements involved in mass transport. Completed with mineralogical studies.
  - Determination of pore size distribution (MIP), specific surface area (BET), and gas and water permeability of specific samples.
  - Through-Diffusion tests with conservative elements (HTO and Cl) to determine the porosity changes in cement (CEM I and/or CEM V) associated to chemical perturbations, with special focus on their possible effects on RN transport. The impact on solid porosity of groundwater changes in: pH, redox, sulphates, carbonates and calcium will be analysed.
- Task 1.3: Conceptual modeling and data interpretation (month 38 to 48)
  - Data interpretation based on the up-scaling (spatial and temporal) of geochemical processes and transport phenomena at the interface scale (+ OPC) and the extent of the altered zone.

12/07/2015

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## WP1: # 6 TUDelft Contribution

Prepared by: Denis Bykov. Effort: 17 Person-months

### Overall objectives:

- Investigate pore evolution and the impact of processes at the interface between Boom clay and high-pH concrete on chemical and physical properties, in relation to Dutch and Belgian disposal concepts.
- Propose theoretical models for designing the experiments and interpretation of the obtained results (in collaboration with NRG in Petten).

### Available Materials/Samples:

- CEM III/B 42.5 LH HS (concrete) for containment of waste (*high pH, fresh and aged interfaces (up to two years)*), low hydration heat, high sulphate resistance)
- CEM I (foamed concrete) for enclosure of waste in the deep geological repository
- CEM III/B 42.5 LH HS (foamed concrete)
- Boom clay (unoxidized)

### Any Deviations from Original Project Plan?:

- None

12/07/2015

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## WP1: # 6 TUDelft, continued

### Description of Work to be Performed/Tasks:

- Task 1.1: Establishing known State-of-the-Art, defining boundary conditions for experimental studies and details of analytical methods for sample characterization (2 PM).
- Task 1.2: Characterization & experimental studies (12 PM).
  - 1.2.1. Studies of the diffusive transport of macro elements (Ca, Mg, S, C) through the interfaces based on the nuclide transport experiments. The aim is to monitor changes that could influence the pore structure in the long term.
    - 1.2.1.1 The accumulation/dissolution of phases containing Ca, Mg, S, C near the interface will be studied and kinetic models will be developed. The migration will be accelerated by using an electro diffusion cell. Kinetic parameters of the processes as a function of concentration, pH, electric potential will be investigated.
    - 1.2.1.2 Characterization of water mobility in the pores will be performed by neutron imaging with the use of deuterium water (or tritium-labeled water).
    - 1.2.1.3 Characterization of concentration profiles of stable elements at the interfaces will be done by neutron activation analysis or radiometrically.
  - 1.2.2. Analysis of pore structure at interfaces: X-ray and neutron imaging (at microscale) + positron annihilation (at nanoscale).
- Task 1.3: Conceptual modelling and data interpretation (3PM).
 

Interpretation of the results obtained in Task 1.2 and using of this data as an input for developing and testing state-of-the art numerical model for processes at cement-clay interface (in collaboration with CEBAMA partner from WP3 - NRG).

12/07/2015

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## WP1: #10 RWMC Contribution



Prepared by: Hitoshi Owada

Effort: 12 Person-months

### Overall objectives:

- To obtain the realistic data for the expansion of altered zone around the contact between cementitious and bentonitic materials.
- To obtain the fundamental data of change of mechanical properties arose from the chemical alteration. Because mechanical and Hydraulic characteristics of bentonitic materials are modeled as functions of effective montmorillonite density, i.e. content of montmorillonite and dry-density of that material, dissolution and/or precipitation of montmorillonite and secondary minerals are important data.

### Available Materials/Samples:

- C-B coupled immersed small specimen.
  - Immersed at RT (around 20 C)
  - Cement type is mainly OPC, almost the same as CEM I in European standard, or 30% fly ash mixed cement (60% W/C ratio).
  - Bentonite is Kunigel V1 (dry density=1.6 or 1.2Mg/m<sup>3</sup>) or MX80 (dry density=1.6 or 1.2Mg/m<sup>3</sup>, only coupled with OPC)
- OPC/Fly ash mixed cement (pH=12.5 or around 11.5)
- bentonitic materials (Kunigel or MX80)

### Any Deviations from Original Project Plan?:

- Mechanical properties such as porosity-compaction relation have already been obtained. Results of those experiments can be shared in CEBAMA project partners.
- Because RWMC's budget is included in that of Japanese national project, RWMC's plan might have some deviations "year by year" by the condition of the contract with Japanese government.

12/07/2015

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## WP1: #10 RWMC, continued



### Description of Work to be Performed/Tasks:

- Ca-XAFS observation of long-term immersed C-B coupled samples and/or artificial analogue specimen. (4-30months)
  - This experiment will be performed till FY2017 and 1 or 2 specimen per year because of the limitation of the machine time of synchrotron-XAFS at High Energy Accelerator Research Organization (72 to 96hrs/year).
  - OPC-Kunigel interface boring core of the formally GMT in Grimsel and 11 years immersed (at RT) OPC or Fly ash mixed cement - bentonite (Kunigel or MX80) coupled specimen that is similar to the diffusion test.
  - Immersion tests carried out around room temperature (20 to 25 C) .
- Measurement of generation and growth ratio of secondary zeolitic minerals (0-32months)
  - By using the Phase-Shift-Interferometer and Atomic Force Microscopy or other techniques.
  - Clinoptilolite and analcime, under compacted and alkaline (pH~12.5) condition at 70 C.
- Change of swelling pressure of montmorillonite during the change from Na-type into Ca-type (0-12months)
  - RWMC already started some experiments to obtain such changes of mechanical properties in the last fiscal year.
  - Flow through condition with CaCl<sub>2</sub> solution at room temperature.

12/07/2015

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## WP1: #11 SCK•CEN Contribution

Prepared by: Norbert Maes. Effort: 18 Person-months

### Overall objectives:

Characterisation of the interface Boom Clay/High pH concrete in terms of changes in transport properties (permeability and diffusion) and microstructure (clogging or opening of pores) due to Ca-leaching and carbonation.

### Available Materials/Samples:

- Different time scales:
  - long term in-situ: HADES, 12 yrs, low Permeability/high pH concrete
  - short term lab: controlled conditions, months, low strength/high-porosity/high pH cement
- In strong collaboration with BRGM: mineralogical and microstructural changes
- HZDR will be involved in analysing transport pathways in fresh and altered cement samples (selection) by means of PET measurements
- Gathered data will serve as input for WP3 (modelling by SCK•CEN and others)

12/07/2015

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## WP1: (#11 SCK•CEN), continued

### Description of Work to be Performed/Tasks:

- Study of existing interface Boom Clay/concrete (HADES – 12 yrs contact, low Permeability/high pH concrete) – Y1-2
  - Coring in HADES + Transport properties (SCK•CEN)
  - $\mu$ -structural analysis (BRGM) of concrete&clay– see input BRGM
  - *Outcome: transport parameters and associated microstructural parameters*
- Study of interface Boom Clay/cement under different controlled conditions – lab (low strength/high-porosity/high pH cement) - Y1-4
  - Batch type experiments:  $f(t)$  interaction of cement plug with Boom Clay suspensions --> changes in chemistry and  $\mu$ -structure (@BRGM, selection of samples)
  - Percolation/column type experiments:  $f(t)$  follow up of transport properties (permeability, diffusivity), changes in  $\mu$ -structure of cement&clay(@BRGM, selection of samples)
  - HZDR will be involved in analysing transport pathways in a selection of fresh and altered cement samples by means of PET measurements
  - *Outcome: transport parameters and associated microstructural parameters*
- Data will serve as input for WP3 (modelling by SCK•CEN and others)

12/07/2015

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## WP1: #13 UJV Contribution

Prepared by: Vaclava Havlova. Effort: 18,5 Person-months

### Overall objectives:

- to study influence of mutual interaction between cement based materials (OPC, LPC) and bentonite (Czech Ca-Mg bentonite) on properties of both materials
- to look into influence on chemical and transport properties
- to study samples that had been in the mutual contact for different period time under different conditions (lab conditions, real system conditions)
- to provide data for modellers

### Available Materials/Samples:

- OPC and Czech Ca-Mg bentonite, both aged for 60 month in Josef underground laboratory (potential exchange of small amount)
- OPC and Czech Ca-Mg bentonite, both aged for 10 – 30 M under lab conditions (potential exchange of small amount)

### Any Deviations from Original Project Plan?:

No

12/07/2015

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## WP1: #13 UJV Contribution

### Description of Work to be Performed/Tasks:

- Close colabration with CTU, potentially VTT
- Task 1.1: Establishing known State-of-the-Art (02/2016)
  - defining boundary conditions for experimental studies and details of analytical methods for sample characterization
- Task 1.2: Characterization & experimental studies (05/2019)
  - Analyses of in-situ samples (bentonite, OPC) after long term contact (up to 60 month): XRD (mineralogical analyses), CEC and cation availability on exchaengeable sites
  - Analyses of reference samples of materials used (bentonite, OPC, LPC if available): XRD (mineralogical analyses), CEC and cation availability on exchaengeable sites
  - Analyses of laboratory samples (bentonite, OPC, LPC if available) after ageing procedure (9/20/30 months): XRD (mineralogical analyses), CEC and cation availability on exchangeable sites
  - Preparation of aged samples for diffusion studies (bentonite, OPC, , LPC if available)
  - Diffusion study on bentonite/cement (OPC, LPC if available) material property changes after mutual interaction:  $^3\text{H}$ ,  $^{36}\text{Cl}$  as conservative tracers will be used
- Task 1.3: Data evaluation and interpretation (11/2017 – 05/2019)
  - Evaluation of gained data, inputs to WP3 (in cooperation with CTU and potentially other CEBAMA partners)

12/07/2015

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## WP1: #15 Uni Loughborough Contribution

Prepared by: Prof Read & Dr Felipe-Sotelo. Effort: 22 months

### Overall objectives:

- Improved understanding of the changes that occur at mineralogical/geochemical interfaces between cementitious materials and groundwater as a function of key environmental parameters

### Available Materials/Samples:

- The cements will comprise a low strength, high pH backfill (NRVB - UK) and two low pH cements (silica fume - Finland & Sweden and high alumina cement, HAC - France).
- The groundwaters will be chosen to be representative of the crystalline Borrowdale Volcanic Group (BVG) and Corallian-Oxfordian Clay (COx).
- The effect of evolving mineralogy on porosity, (water and gas) permeability and potential bulk transport properties will be investigated by techniques including gas porosimetry,  $^{14}\text{C}$ -PMMA/autoradiography, porewater squeezing and X-rayCT.

### Any Deviations from Original Project Plan?:

- Not expected

12/07/2015

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## WP1: Uni Loughborough, continued

### Description of Work to be Performed/Tasks:

- Phase 1 (6 months)
  - Curing of target cements at ambient temperatures, variable gas pressure and water saturation.
- Phase 2 (36 months)
  - Experiments using synthetic groundwaters to investigate alteration processes and products at cement-(simulated ground) water interfaces.
  - Preparation of cement samples in solutions at two ionic strengths, approximating fresh groundwater and seawater.
  - Investigation of the impact of different curing regimes (salinity, controlled carbonation) on evolving mineralogy at the cement-groundwater interfaces.
  - Periodic solution analysis by ICP-MS, ICP-OES and ion chromatography to obtain compositional data for modelling purposes.
  - Mineralogical and textural characterisation of samples: Characterisation of solids by ( $\mu$ )XRD, ( $\mu$ )XRF, ESEM, TEM.
- Phase 3 (6 months)
  - synthesis, provision of data for WP3 and final reporting.

12/07/2015

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## WP1: # 16 CTU Contribution

Prepared by: Lucie Hausmannova. Effort: 22 Person-months

### Overall objectives:

- Experimental study on effects of mutual interaction between cement based materials (OPC, LPH) and Czech Ca-Mg bentonite
- Testing of “new” samples after 9 to 27 months of cement-bentonite interaction under defined conditions (high temperature,...). “Old” samples available from previous experiment (testing “cartridges” at Josef URL since 2010) will also be tested.
- Laboratory tests on cement: strength properties
- Laboratory tests on bentonite: Hydraulic conductivity, swelling pressure, liquid limit, swell index and retention curves
- Providing samples to UJV Řež for geochemical testing

**Available Materials/Samples:** NO

**Any Deviations from Original Project Plan?:** NO

12/07/2015

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## WP1: CTU continued

### Description of Work to be Performed/Tasks:

- Task 1.1: Establishing known State-of-the-Art. defining boundary conditions for experimental studies and details of analytical methods for sample characterization (02/2016)
  - Outcome: Test conditions defined
- Task 1.2: Characterization & experimental studies (05/2019)
  - Outcome: Study on effect of long term interaction between cement based materials and bentonite - influence on both bentonite and cement behaviour (hydro physical and mechanical prop.)
  - Verification of non-standard strength tests methods to be used on thin samples (esp. correlation relationships)
  - Running of “ageing procedures”, sampling and evaluation after 9/18/27 months, providing samples to UJV Řež, **materials:** Czech bentonite B75, OPC 42.5, LPH cement from VTT
  - Evaluation of the in-situ samples from Josef gallery (since 2010), providing samples to UJV Řež, **materials:** Czech bentonite B75, OPC 42.5
- Task 1.3: Conceptual modelling and data interpretation (11/2017 – 05/2019)
  - Outcome: Evaluation of gained data, inputs to WP3 (in cooperation with UJV Řež and potentially other CEBAMA partners)

12/07/2015

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## WP1: # 17 USFD Contribution

Prepared by: Claire Corkhill. Effort: 42 Person-months

### Overall objectives:

- USFD will undertake a detailed physico-chemical characterisation of selected cements relevant to international GDF concepts
- Build a mechanistic understanding of how cement / ground water interactions are likely to influence diffusive transport
- Advance understanding of porosity, permeability and cement mineral phase assemblages as a function of carbonation, pH, salinity and ground water composition.

### Available Materials/Samples:

- Low strength, high-pH cement (UK Nirex Reference Vault Backfill)
- Low-pH (PC-silica fume)
- Low-pH blend (PC-silica fume-fly ash)
- Cured under ambient conditions for: 28d to 1 year, 2 years and 3 years.
- Subset of samples cured under controlled CO<sub>2</sub> conditions
- Samples will be immersed in ground water solutions (granitic, clay and seawater)

12/07/2015

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## WP1: (USFD), continued

### Description of Work to be Performed/Tasks:

- **Task 1.1.** Publishable literature survey on UK GDF cement interfaces. Months 1 - 12.
- **Task 1.2.** Mechanistic understanding of physical processes governing interfacial groundwater / cement reactions. Months 10 – 36.
  - Identify changes in porosity, permeability and tortuosity as a function of alteration by groundwater
  - Physical characterisation using MIP, gas adsorption, oxygen permeametry,  $\mu$ -XCT, neutron tomography, SEM and TEM.
  - USFD will also support Ullough in their chemical characterisation of the same cements, using NMR and Rietveld refinement of X-ray Diffraction data.
- **Task 1.3.** Develop a quantitative model of how cement / groundwater interactions affect diffusive transport. Months 37 – 42.
  - Datasets from Task 1.2. will be combined and investigated through application of diffusive transport modelling.
  - The USFD student will visit is CEBAMA WP3 partner to maximise the outcomes of this task.

12/07/2015

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## WP1: # 18 – VTT Contribution

Prepared by: Tapio Vehmas, Erika Holt. Effort: 30 Person-months

### Overall objectives:

- Studying cementitious materials degradation in different groundwater/clay environment.
- Estimation of alkaline plume in various leaching scenarios.
- Establishing time dependent behaviour of Low-pH materials behaviour in various conditions.
- Work is conducted in **close cooperation with Posiva**. Low-pH recipes (grout, mortar, concrete) supplied and results reported to them.
- Possible partner cooperation with UJV/CTU, CSIC, Andra

### Available Materials/Samples:

- Batch samples of CEM I/silica/fly ash in various degrees of pozzolanic reaction.
- Equilibrium batch samples in saline/distilled/bentonite aqueous solutions.

### Any Deviations from Original Project Plan?:

- none

12/07/2015

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## WP1: VTT, continued

### Description of Work to be Performed/Tasks:

- Literature review and state-of-the-art, for parameters needed (Months 0-9)
  - equilibrium compositions of calcium-silicate-hydrate gels in aqueous solutions.
  - calcium-silicate-hydrates /ion –interactions
  - design of experimental mixtures, test conditions (inputs to D1.02-1.04)
- Experimental Work (Months 6-36)
  - Equilibrium studies for:
    - Determination of congruent dissolution of various binder, CSH and leachate compositions. Leachate pH and composition analysis (ICP)
    - Characterization of solid phase composition (XRD, TG) polymerization degree (Si-NMR) and Ca : Si/Al – ratio (EDS).
  - Kinetic studies on:
    - Rate of pozzolanic reaction at various stages. Matured and accelerated samples, various temperatures & fineness, probably activation energy –based model. Planned methods: conduction calorimeter, XRD and TG.
    - Leaching effect and chemical composition of leachate. Leachate pH and composition analysis (ICP), groundwater interaction. Methods SEM, EDS, thin section analysis, XRF
    - Incongruent dissolution in above presented scenarios. Determination of leachate flow rate effect.
- Conceptual Modeling & data interpretation (Months 24-46)

12/07/2015

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## WP1: # 19 HZDR Contribution

Prepared by: Johannes Kulenkampff. Effort: 5 Person-months

### Overall objectives:

- Provide spatio-temporal positron emission tomograms (PET) of transport processes as 4D reference data sets for numerical modelling.
- Quantification of changes of effective porosity and permeability by correlated PET/CT interpretation.
- Enhanced process understanding of the interactions at interfaces between cementitious and clay formations, by applying the unique non-invasive GeoPET method in joint experiments with SCK CEN and UniBE.

### Available Materials/Samples:

- Boom clay from SCK CEN
- Opalinus clay from UniBE

### Any Deviations from Original Project Plan?:

- No

12/07/2015

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## WP1: HZDR, continued

### Description of Work to be Performed/Tasks:

- Design of experiments, sample preparation
  - PET provides non-destructively spatio-temporal quantitative tracer concentrations with highest sensitivity over an integration volume (voxel size) of 1  $\mu\text{L}$  – ideal for process monitoring (flow and diffusion) including alterations of the material on core scale. Structural information is gained with  $\mu\text{CT}$ .
  - The experiments will be designed in close collaboration with the partners SCK CEN and UniBE.
- Degradation of cement with Boom-clay pore water
  - Percolation of Boom clay pore water through freshly manufactured cement samples.
  - Visualization over time of the propagating fluids with PET, analysis of the pore water composition.
  - Together with SCK CEN.
- Degradation of aged cement/claystone and cement/bentonite interfaces
  - Propagation monitoring of reacting fluids propagating (slow advection and diffusion) through aged cement/Opalinus Clay and cement/MX-80 bentonite interface from the Mt Terri URL.
  - Together with UniBE.
- Evaluation and modelling
  - Parameter determination for the processes to be quantified: Migration of injected fluids, clogging or opening of transport effective pores in cement component (effective volume, propagation velocities, diffusion coefficient ..)
  - Supply of experimental reference data for model verification and calibration in WP3 (SCK CEN, UniBE, PSI).

12/07/2015

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## WP1: # 20 LML Contribution

Prepared by: Jianfu SHAO. Effort: 30 Person-months

### Overall objectives:

- Develop a series of laboratory tests for the characterization of hydromechanical behavior of the concrete/argillite interface under complex mechanical, hydraulic, hydric, thermal and chemical conditions;
- Provide sound experimental data for numerical modeling in WP3,

### Available Materials/Samples:

- Cox argillite - low pH concrete interfaces
- Close cooperation with Andra on the choice of sample geometry and testing conditions
- A room-designed specific device for direct shear tests of interfaces
- A high resolution micro-tomography for micro-structural analysis of interfaces

### Any Deviations from Original Project Plan?:

- None

12/07/2015

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## WP1: LML, continued

### Description of Work to be Performed/Tasks:

- State-of-the-Art (0-6 months)
  - An extensive synthesis on the mechanical properties and permeability of concrete/argillite interfaces;
  - A particular attention on the effects of saturation, temperature and carbonation process;
  - Choice of sample geometry, boundary conditions for both mechanical and hydromechanical tests, value ranges of temperature, saturation degree and carbonation process
  - 0 – 6 months
- Characterization and experimental studies (6-36 months)
  - Four groups of laboratory tests
    - Mechanical test under room temperature (20°C) and saturated condition (RH=100%),
    - Hydromechanical tests under 20°C and 100%RH
    - Influences of temperature (40°C, 60° and 80°C) and relative humidity ( 50%, 70% and 90%)
    - Effects of chemical degradation (carbonation of low pH concrete)
- Conceptual modelling and data interpretation (24-48 months)
  - Conceptual modeling and data interpretation
  - Failure criterion of the interface under combined effective normal stress and shear stress
  - Criterion for the occurrence of normal dilatancy (shear induced opening) of the interface
  - Relationship between the permeability, effective normal stress and normal strain (interface closure and opening) of the interface
  - Impacts of temperature, relative humidity and carbonation process

12/07/2015

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## WP1: #21 UAM Contribution

Prepared by: Jaime Cuevas. Effort: 24 Person-months

### Overall objectives:

- To perform the geochemical study of **high pH** concrete (OPC)-FEBEX-bentonite at in-situ and long-term experiments
- To determine the characteristic surface interface reactivity of compacted FEBEX bentonite induced by OPC based concrete pore water (high and low pH)

### Available Materials/Samples:

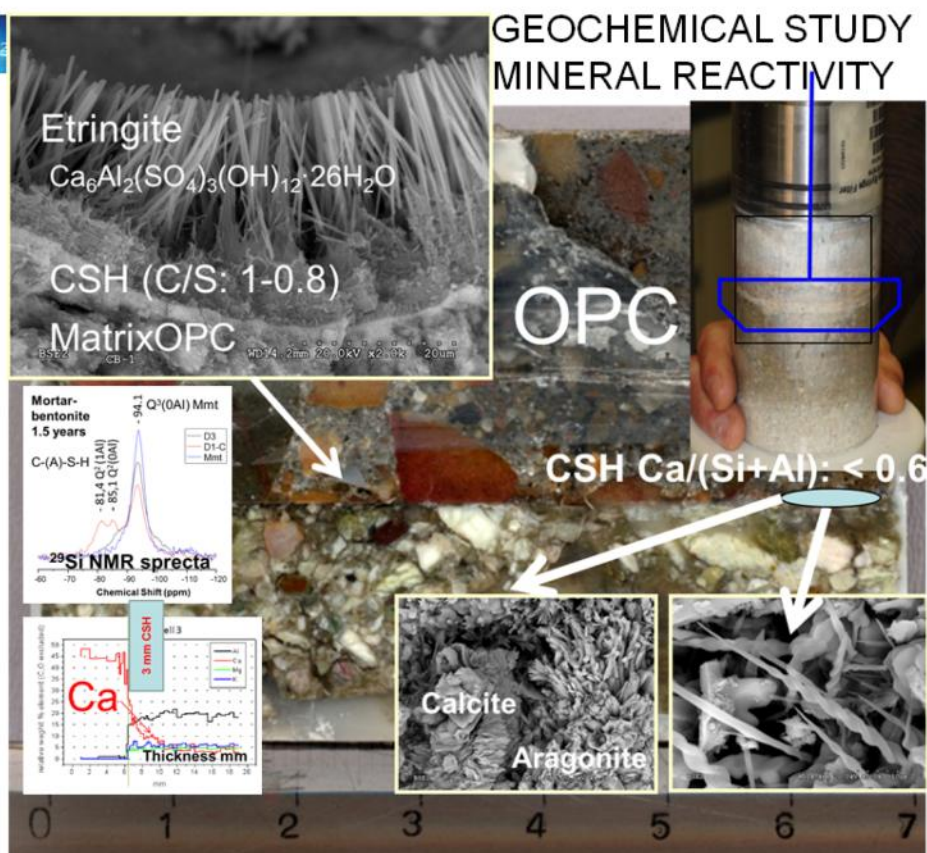
- FEBEX bentonite in contact with OPC from the HB6 laboratory experiment lasted 10 years. **Samples shared with CIEMAT and CSIC.**
- FEBEX bentonite in contact with OPC from the in situ FEBEX experiment (GTS).. **Samples shared with CIEMAT and CSIC.**
- Small size high and low pH (OPC based) concrete-FEBEX bentonite interface experiments (OPC: CSIC)

### Any Deviations from Original Project Plan?:

- No

12/07/2015

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## WP1: UAM, continued

### Description of Work to be Performed/Tasks:

- **Task 1.1: State-of-the-Art, defining concepts (upscaling) and details of analytical methods for sample characterization**

Sampling and conditioning of in situ large scale at **OPC** interfaces (**FEBEX**):

(1-6 months)

Design and implementation of SurfaceE Reactivity Interface Experiments (**SERIE**)

(1-6 months)

- **Task 1.2: Characterization & experimental studies.**

- **1.2.2. Interfaces with bentonite**

Characterization of FEBEX interfaces (6-42 months)

Sequential sampling and characterization of **SERIE** (6-30 months)

Dismantling of **HB6** cell (10 years): sub-sampling and characterization (12-38 month)

- **Task 1.3: Conceptual modeling and data interpretation.**

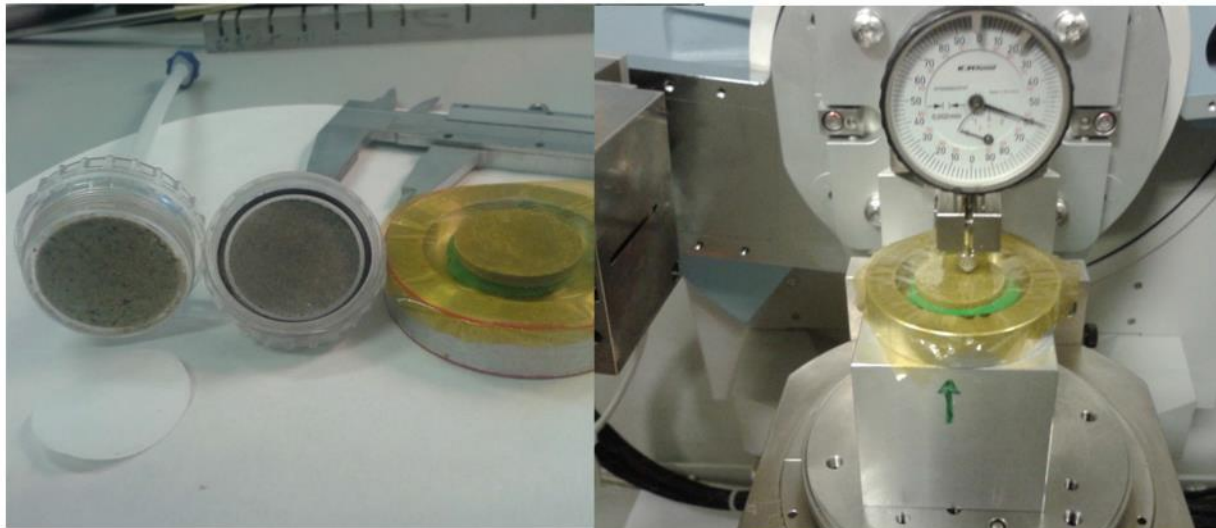
Complementary characterization of interfaces (30-42 months)

Integration for local to global (SERIE-HB6-in-SITU) geochemistry. Inputs for model validation (38-46 months).

Conclusions for performance of concrete-FEBEX interfaces (46-48 months)

12/07/2015

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## WP1: #22 CSIC Contribution

Prepared by: María Cruz Alonso. Effort: 24 Person-months

### Overall objectives:

- Evaluation of the long-term aging of high-pH concrete (100%OPC) alteration from in-situ large-medium scale experiments (FEBEX in-situ experiment, 13y & HB6 cell, 10y) in contact with different EBS (Concrete/FEBEX bentonite & Concrete/granite interfaces) to identify the influence on transport properties due to porosity concrete alteration.
- Assessment of the impact of EBS granitic ground waters on transport properties of concrete (high and low-pH) from chemical modifications of solid phases to physical alteration properties affecting porosity after short-term ages of contact.

### Available Materials/Samples:

- High pH concrete cores from in-situ large scale long-term experiments: *available end 2015-2016. Samples shared: CIEMAT/UAM/CSIC*
- High/low pH concrete production: *designed and fabricated by CSIC. Samples shared CSIC-UAM*

### Any Deviations from Original Project Plan?:

- None

12/07/2015

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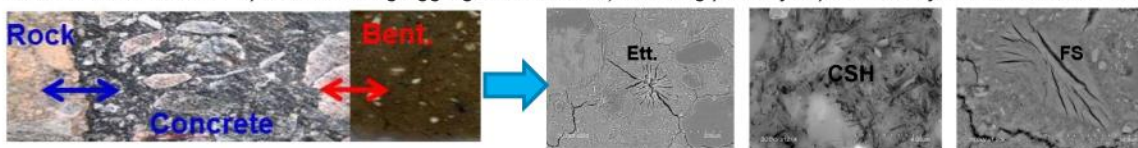
### Description of Work to be Performed/Tasks:

- **Task 1.1: State-of-the-Art, defining boundary conditions for experimental studies and details of analytical methods for sample characterization. (1-6M)**

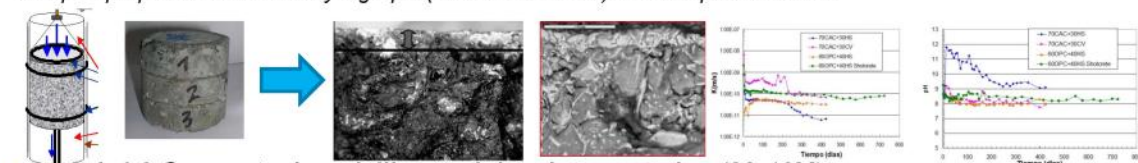
Implementation of the testing procedures and identification of main parameters influencing the concrete aging, at long-term & at short-term, for (high & low pH) that alter concrete transport properties due to the interaction with EBS.

- **Task 1.2: Characterization & experimental studies. (6-42M)**

1) Long-term degradation of concrete: Evaluation of aging alteration of high-pH concrete in the different EBS interfaces from in-situ large/medium scale long-term experiments, in terms of: hydrates alteration (chemical or mineralogical modifications of cement paste including aggregate interfaces) affecting porosity & permeability of the concrete matrix.



2) Short-term interaction concrete/EBS: Assessment of the impact of different granitic ground-waters from EBS on transport properties of laboratory high-pH (similar to FEBEX) and low-pH concretes.



- **Task 1.3 Conceptual modelling and data interpretation (38-46M).**

Integration & up-scaling of the obtained results & conclusions for physical-chemical performance of concretes in contact with different EBS

12/07/2015

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## WP1: # 23 - Andra Contribution

Prepared by: X. BOURBON. Effort: 11 person-months

### Overall objectives:

- THM evolution during setting and delayed HM properties evolution with time at Clay/low pH concrete interface
- Atmospheric carbonation of low pH concrete in contact with ventilation air
- Application to concrete wall evolution during the operating period
- Mineralogical evolution at low pH concrete/clay host rock interface

### Available Materials/Samples:

- Low pH concrete cast in the French URL (two formulations)
- Concrete/clay host rock interfaces ; in situ test (scale 1) including operating condition

### Any Deviations from Original Project Plan?:

- none

12/07/2015

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## WP1: Andra, continued

### Description of Work to be Performed/Tasks:

- Design and set up (0-9 months)
  - Design of the experiment, including :
    - Formulations of low hydration heat/low pH concrete
    - Choice of the sensors
  - Installation
- Data acquisition / Experimental studies (10-36 months)
  - Field test in the French URL (Bure)
  - THM data acquisition with time in operating conditions (thick walls in an open gallery ; ~20°C / RH 40/60%)
- Interpretation (37-48 months)
  - THM Model validation for low pH concrete at a 'representative' scale
  - Validation of the input data with regards to the physical behaviour (hydration, drying, shrinkage, ...)
  - Chemical evolution at the interfaces : impact of atmospheric condition on low pH concrete behaviour (incl. carbonation) ; impact of the host rock properties on the physical and chemical behaviour

12/07/2015

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## WP1: # 25 UNIBERN Contribution

Prepared by: Urs Mäder. Effort: 44 Person-months

### Overall objectives:

- Perform long-term transport experiments on 3-7 year-old interfaces of claystone/concrete.
- Observe stationary phase (X-ray CT) as well as mobile phase, and carry out detailed post-mortem characterization.
- A complete description of the state and time-evolution of a realistic interface system is attempted with inferred consequences for radionuclide transport.

### Available Materials/Samples:

- Aged (3a, 7a) samples from Mont Terri URL (CI Project): Opalinus Clay in contact with OPC mortar/paste/concrete and ESDRED (low-pH) mortar/paste/concrete

### Any Deviations from Original Project Plan?:

- 1 month delay in effective start of project due to initially uncertain Swiss-EU funding situation

12/07/2015

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## WP1: (UNIBERN), continued

### Description of Work to be Performed/Tasks:

- Task 1.1: State-of-the-art and boundary cond. for experiments (M 1-7)
  - Focus on concrete-claystone interface experiments performed in-situ, and how to transfer boundary conditions into a laboratory set-up
  - Identification of geochemical processes and physic-chemical evolution observed in in-situ experiments of concrete-claystone interaction; Experimental boundary conditions for experiments and choice of analytical methods.
- Task 1.2: Transport experiments: interfaces with claystone (M 6-42)
  - Set-up and carry out 3 confined transport experiments, one in collaboration with HZDR
  - Experiments on concrete/clay interfaces aged over 3-7 years in a realistic environment (e.g. Mont Terri in-situ experiment) will give directly relevant transport parameters
- Task 1.3: Conceptual modelling and data interpretation (M 30-48)
  - Interpret semi-quantitatively data from bulk, 2D and 3D characterization of stationary phase and mobile phase in terms of processes and transport properties.
  - Complete description of stationary and mobile phase and its interpretation will provide comprehensive data set for reactive transport modelling interpretation (WP3).

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## WP1: # 26 IRSN Contribution

Prepared by: A. Dauzeres. Effort: 37 Person-months

### Overall objectives:

- (Task 1-1) Establish a State-of-the-Art concerning the geochemical evolution of cementitious materials placed in clayey environment between 50°C and 80°C
- (Task 1-2) Improve understanding of chemical processes influence at the clay/concrete interfaces on the porosity evolutions at 70°C. Focus on new cementitious materials (low pH binary mix).

### Available Materials/Samples:

- 6 in situ tests are already installed in the Tournemire URL at 70°C (CEMTEX program) : Toarcian argillites/concretes interfaces (*low pH and OPC, possibility to exchange samples with other partners*)
- 4 tests (2 with OPC and 2 with low pH) already totally or partially characterized after 1 and 2 years of interaction. Last 2 tests will be extracted in 2017 (i.e. after 5 years of interaction)

### Any Deviations from Original Project Plan?:

- No

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## WP1: (IRSN), continued

### Description of Work to be Performed/Tasks:

- Design and implement 3 new in situ tests with a low-pH formulation, representative of the French concept (0-6 months)
  - Modification on the initial heating device, building of the device, installation of the recording system, sensors...
  - Needs of the new low pH formulation (Andra) – cement pastes preparation
  - Start of the 3 tests in January 2016
- Tests follow-up (7-42 months)
  - Control of the thermal regulation near the interface in the material
  - Control of the constant full water saturation conditions
- Sampling and characterizations (18 months – 48 months)
  - Test 1 year – end January 2017
  - Test 2 years – end January 2018
  - Test 3 years – end January 2019
  - Characterizations of mineralogy and microstructure evolutions (collaboration with the HYRL (Finland))

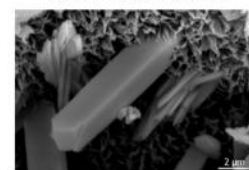
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CEMTEX experiment



20 cm  
Device cross-section



SEM picture of interface

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