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"Full Scale Demonstration of Plugs and Seals"

DOPAS - Work Package 3

Deliverable D3.8 Test report on FSS cast in box concrete

with low pH concrete formulas for FSS and Laboratory work on the performance of low pH concrete for FSS

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ABSTRACT:

This report combines information on low pH SCC development into the one Deliverable D3.8 including information originally planned for D3.4 Report on low-pH concrete formulas for FSS; D3.6 Lab report on the performance of low-p H concrete for FSS and D3.8 Test report on FSS cast in-box concrete.

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Executive Summary

In DOPAS, Work Package 3 (WP3) is related to the construction of large scale demonstrators of seals and plugs.

FSS (Full-scale Seal) is the seal demonstrator built by Andra with the scientific help of NAGRA, while other WMO's (DOPAS partners) are building or have built their own prototype.

This report gives an overview of the development work carried out in lab and later validated in field (via metric scale tests) to develop, select and validate the formula of the low pH SCC used for the construction of the first component of the FSS seal, i.e. the upstream containment wall (constructed in July 2013).

The formula selected was also used for the casting of the concrete blocks to be used in the future construction of the support wall separating the second FSS component, i.e. the bentonite core from the third FSS component, i.e. the downstream low pH shotcrete containment wall.

The document includes an overview of the main lab and field tests (illustrated by photos) c/w practical details and measures.

Links to previous and future Andra's FSS specific (or DOPAS more general) deliverable reports are also given.

List of Acronyms

This generic list of acronyms concerns entities, activities, concepts, equipment and materials which are Andra (or DOPAS Partners) specific in the context of the FSS experiment.

ASN: Autorité de Sûreté Nucléaire (Nuclear Authority).

CIGEO: Centre Industriel de Stockage Géologique (Industrial Repository, AKA Cigéo).

CEA-LECBA: Company contracted for the low pH concrete mixes formulation and qualification (a member of the GMES Consortium)

CNE: Commission nationale d'évaluation (National Assessment Board).

CSH: Calcium Silicate Hydrates.

DGR: Deep Geological Repository (see also GDF)

DOPAS: Full-scale Demonstration of Plugs and Seals (Name of Project on Seals).

EBS: Engineered Barrier System.

EC: European Commission.

EDZ: Excavation damaged zone.

ESDRED: Engineering Studies and Demonstration of Repository Designs (name of previous EC supported Project).

FSS: Full-scale Seal.

GDF: Geological Disposal Facility.

GME: Groupement momentané d'entreprises (FSS General Contractor formed as a Consortium of companies).

HLW: High-level Waste.

IAEA: International Atomic Energy Agency.

ILW: Intermediate-level Waste.

IRSN: Institut de Recherche sur la Sûreté Nucléaire (Expert Organization support to ASN).

LLW: Low-level Waste.

OPC: Ordinary Portland cement.

RA: Concrete hardening Retarder

R&D: Research and Development.

SCC: Self-compacting concrete or self-consolidating cement.

SP: Concrete Super-Plasticizer.

SMC: Supplementary cementing materials

URL: Underground research laboratory (Bure is the French URL).

WMO: Waste Management Organization.

WP: Work Package.

List of DOPAS Project Partners

The 14 partners (from 9 countries) in the EC supported DOPAS Project are listed below. In the remainder of this report each partner (if mentioned) is referred to as indicated:

Andra:	Agence nationale pour la gestion des déchets radioactifs (France).
B+ Tech:	B+ Tech Oy (Finland).
CTU:	Czech Technical University (Czech Republic).
DBE TEC:	DBE TECHNOLOGY GmbH (Germany).
GSL:	Galson Sciences Limited (United Kingdom).
GRS:	Gesellschaft für Anlagen und Reaktorsicherheit (Germany).
Nagra:	Die Nationale Genossenschaft für die Lagerung Radioaktiver Abfälle (Switzerland).
NDA:	Nuclear Decommissioning Authority (United Kingdom).
NRG:	Nuclear Research and Consultancy Group (The Netherlands).
Posiva:	Posiva Oy (Finland).
SURAO:	The Radioactive Waste Repository Authority (Czech Republic), aka RAWRA.
SKB:	Svensk Kärnbränslehantering AB (Sweden).
UJV:	UJV Řež a.s. (Czech Republic).
VTT:	Teknologian Tutkimuskeskeskus VTT (Finland).

Table of Contents

1. National Context for the FSS Experiment

In France, the repository host rock is the 155-million-year-old Callovo-Oxfordian clayish formation, which lies in the east of the Parisian Basin. The repository project is referred to as Cigéo. The disposal reference inventory includes long-lived Intermediate-level waste (ILW) from operation, maintenance and decommissioning of nuclear facilities and HLW from spent fuel reprocessing. The waste will be disposed of in physically separated disposal zones: one for ILW and one for HLW. The repository's primary function is to isolate the waste from human activities at the surface and its second function is to confine radioactive substance and control the transfer pathways which may in the long term bring radionuclides into contact with humans and the environment (Andra, 2013). The principal contribution of the seals in Andra's concept is to provide the second function.

The ILW disposal zone includes several tens of large-diameter disposal vaults, each about 500m long. Vault concrete lining and disposal containers provide a cementitious (buffer) environment for the ILW waste. The gaps between waste packages and vault lining could be left empty or backfilled with cementitious material or neutral filler (e.g. sand).

In the French concept, seals are defined as hydraulic components for closure of large diameter (several meters) underground installations and infrastructure components such as shafts, ramps, drifts[1] and ILW disposal vaults. Each seal consists of a swelling clay core (EBS) and concrete containment walls. The conceptual design of drift and ILW disposal vault seals is the same. The location of seals in the planned Cigéo repository is shown in Figure 1.

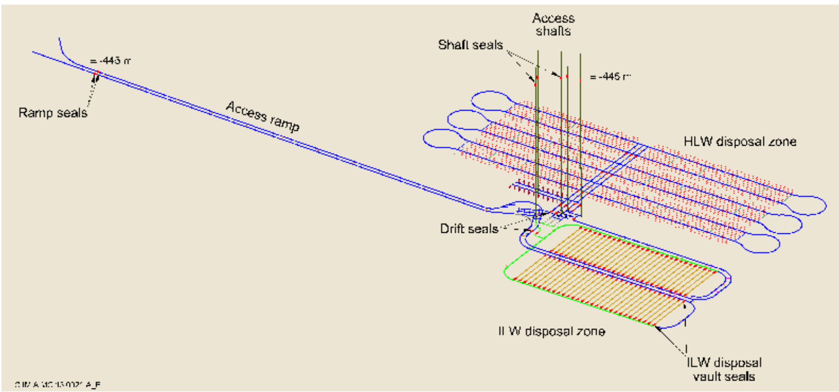


Figure 1: Location of the seals in the French repository concept

[1] Drifts are horizontal tunnels, whereas ramps are inclined tunnels.

2. FSS Design Basis and Link to the Cigéo Reference Design Basis

The FSS experiment is a full-scale technical demonstration of construction feasibility for a drift and ILW disposal vault seal, being carried out in a hangar of a surface facility in Saint-Dizier, which is close to the French URL at Bure.

The FSS test calls for a large excavation, with a significant length and a considerable amount of equipment and materials mobilized and emplaced. The Bure URL is essentially a qualification facility, in which the logistical means are somehow limited (transport means, number of people admitted underground, geometry restrictions for large pieces of equipment, etc.). Moreover the Bure URL is busy with various other experiments which cannot be conducted concurrently with large experiments such as FSS.

For that reason, and for standalone reasons, like global experimental costs, global schedule and needs for dismantling, it was decided to go for a surface facility, instead of working underground. The Saint-Dizier site was proposed by the Contractor (GME) in charge of the FSS test, and accepted by Andra, since the vicinity of Bure (30km), the height of hangar (more than 10m of free gap under the roof frame), and the possibility of air parameters control were in line with the experiment technical expectations.

2.1 FSS Design basis

The FSS test is part of a wide-ranging programme of R&D and demonstrator experiments that was established in response to the discussions with ASN and the French National Assessment Board (CNE) in 2009, during which it has been noted that seals, and in particular drift and ILW disposal vault seals, require demonstration in order to achieve licensing authorisation.

As a result, R&D studies and demonstration tests have been launched to assess the technical feasibility and to develop the post-closure requirements of seals in the repository. Those tests cover the performance and constructability issues. FSS belongs to this last category.

The main objective of the FSS test is to develop confidence in, and to demonstrate, the technical feasibility of constructing a full-scale drift (or ILW disposal vault) seal. Technical feasibility includes demonstrating the ability of the approach used to emplace the clay to be suitable for filling recesses (breakouts) in the clay host rock, and also the capacity to build large low pH concrete containment walls with satisfactory mechanical properties.

The FSS test is focused on the construction of the seal, and the swelling clay will not be saturated or otherwise pressurised. The conceptual design of the FSS test is illustrated in Figure 2.

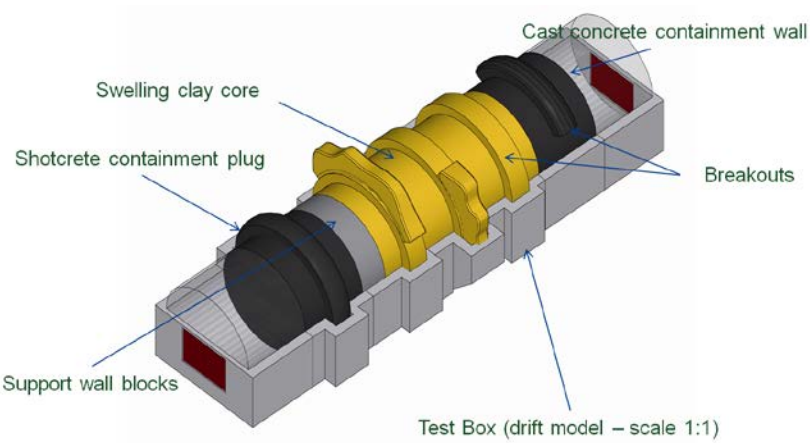


Figure 2: Conceptual design for the ANDRA FSS test.

The main difference between the Cigéo reference and FSS design bases for the Andra drift seal is the length of the seal. The real seal underground will be longer than the seal considered in the FSS experiment. The Cigéo seal design basis and that of the FSS test are justified in DOPAS Deliverable D2.1 "Design Bases and Criteria".

The FSS test box is some 7.6m ID and 36m long. The drift concrete liner (70cm thick) and the formation break outs (recesses) likely to be generated by the drift lining deposition (up to 1m depth at the liner extrados) are simulated.

Representative underground ambient conditions (temperature around 18-30 °C, hygrometry between 50% and 75%, ventilation), have to be maintained within the drift model.

Low pH cast-concrete/shotcrete 5m long containment walls close the volume of the swelling core, on both sides. The bentonite swelling core is some 14m long.

2.2 FSS Test Box Design

The FSS Test Box design was elaborated between July and October 2012. The workshop drawings were also supplied during that period on the basis of the FSS test box concept (as specified by Andra), and (following modelling and dimensioning) derived from the schematic and general lay-out presented in Figure 3.

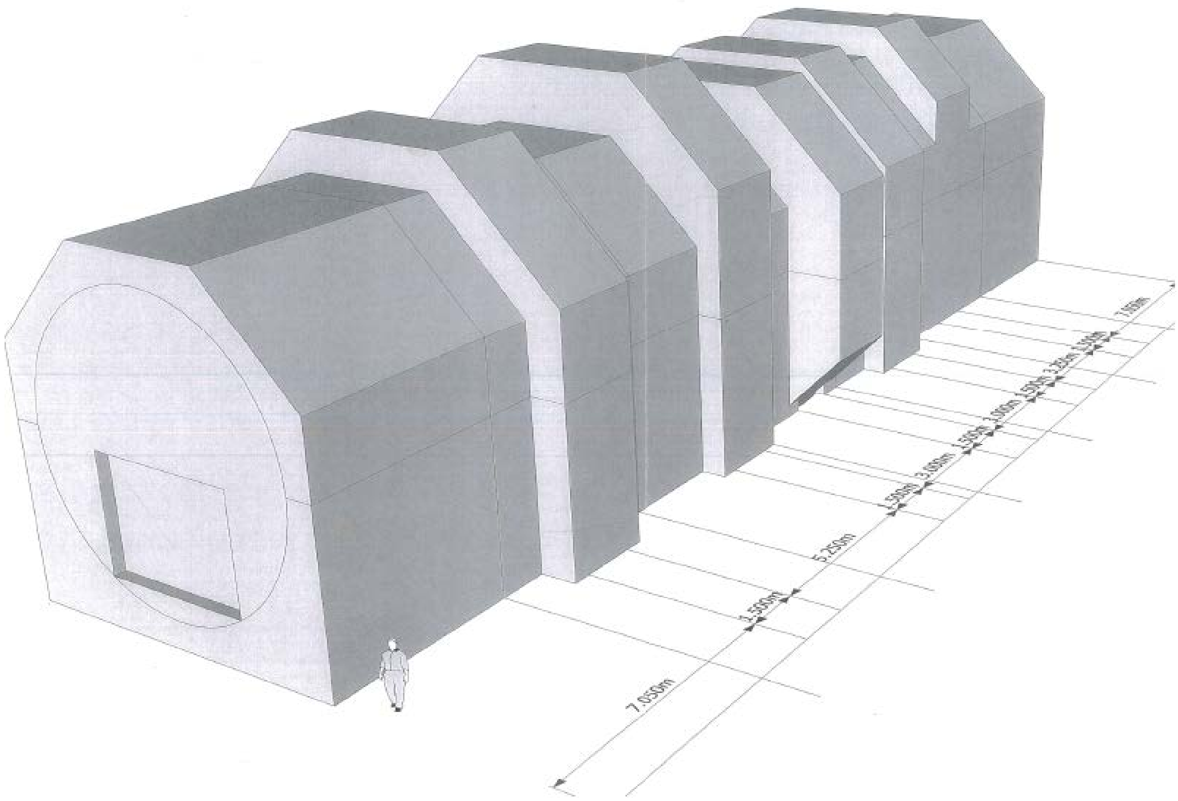


Figure 3: 3D schematic of the FSS test box

3. The Construction of the Test Box

Turning the first sod for the FSS construction took place on October 29th, 2012, with a partial cutting and dismantling of the hangar concrete slab. On December 10th, 2012, the lower part of the concrete box framework was started. Before that a concrete foundation raft had been poured on the newly created platform.

The reinforced concrete box structure was then built with 7 lower blocks and 7 upper blocks (each 5m long). Nine weeks were necessary to build the lower part of the box. Only 13 weeks were necessary to build the upper part and the last block concrete casting phase took place on May 2013.

The access to the top of the test box was made possible thanks to a set of stairs. In order to see and check the bentonite backfill, 12 observations windows were also created. A local exhaust ventilation system ("mine like") was installed, with a closing door in the front of the box, in order to control the ambient temperature and the average hydric rate. A temperature and hygrometry monitoring device was also installed.

The box could then be commissioned and get the "ready for experiment" status (Figure 4).

The Test Box construction story is documented in DOPAS Deliverable D3.10.

Figure 4: The FSS test box ready for seal construction

PS: It can be noted that no special research work was done on the concrete used for the test box construction; it was based on an ordinary Portland cement (OPC) and on common local aggregates.

4. The development of the low pH concrete formulas in lab and in field

4.1 Introduction (problematic and iterative approach):

Low-alkalinity concretes are characterized by the low pH value of their interstitial solution (between 10.5 and 12.2 depending on the binder composition).They are obtained by using large additions of supplementary cementing materials (SCM) to dilute the cement alkalis, convert portlandite into Calcium Silicate Hydrates (CSH) and lower the C/S ratio of the CSH. Many SCM can be used but the key in reducing the pH value is the silica content. As such, silica fume remains the most efficient material of all the SCMs.

Low alkalinity concretes are generally made with binary blends of CEM I (ordinary Portland cement) and silica fume (usually 60% of CEM I + 40% of silica fume). A binary blend of CEM III/B (CEM I with addition of some 66% to 80% of slag) with nanosilica (some 10%) was however studied in Mont Terri laboratory. The CEM III/B was used to limit the formation of portlandite at early age whereas nanosilica was used to increase the hydration rate (on the long term, the pH value reached 12.2). Ternary blends of CEM I (together with slag, silica fume, fly ash or metakaolin) can also be found in literature Using another SCM together with silica fume allows counterbalancing the detrimental effects of silica fume at high level and mitigating the heat emitted during hydration as well as the shrinkage of the resulting low-alkalinity concretes.

The DOPAS-FSS project involves the fabrication of two low-alkalinity concrete plugs: one made with shotcrete and the other with SCC. This chapter addresses the formulation and qualification works related to the low pH SCC, first in lab, then in field at a metric scale.

The FSS SCC concrete requirements (as posted in Andra's Scope of Work) are summarized below:

- The pH value of the pore solution must be lower than 11.0 at 28 days (and ideally between 10.5 and 11.0),
- The SCC must be pumpable and useable two hours after mixing (to account for the time needed to transport the concrete from surface down to 500 m underground in Cigéo).
- The compressive strength must be greater than 30 MPa after 28days and 40 MPa after 90 days respectively.
- The maximal temperature reached within the containment wall (Ø8×5 m) must be less than 50°C at all times.

The design of a proper concrete formulation was dealt with in three consecutive steps:

- First, different binder compositions were tested in lab and the most efficient ones in reducing the pH were then selected for the second phase,
- The second step consisted in proportioning the SCC mix. The resulting concretes were tested in the laboratory and their properties of interest were measured.
- Finally, in a third step, the most promising mixes were tested at a metric scale (in field) using the ready-mix plant selected for the FSS project.

This approach which enabled the selection of the best formulation used further for the full scale construction of the SCC upstream containment wall in FSS is described hereafter.

4.2 Binder Composition

Advantage was taken of the binder compositions previously developed for Andra by CEA-LECBA (a member of the consortium of companies forming the GMES) in previous works (in 2008): one binary mix and two ternary ones were selected (Table1).

The two ternary blends were mixtures of CEM I and fly ash/silica fume (mix T1) or slag/silica fume (mix T3) while the binary blend was a mixture of CEM I and silica fume. The silica fume content of this blend was 40%. But according to the experience so far acquired, this content was deemed not sufficient to meet the pH requirement (i.e. 11.0 at 28 days). The silica fume content was then increased up to 50% and the resulting binder composition was denominated B50 CEM I (i.e. 50% CEM I + 50% silica fume).

Alternatively the experience acquired in Mont Terri Underground Laboratory (in 2012) on a binary blend of CEM III and silica fume was also studied: 10% of silica fume was clearly not sufficient to meet the pH requirement, so three different silica contents ranging from 30% to 50% were studied. The three resulting mixtures (binary blends) were respectively denominated B30, B40 and B50 CEM III (Table 1).

Table 1: Binder compositions

Compounds	Ternary blends		Binary blends			
	T1	T3	B50 CEM I	B30 CEM III	B40 CEM III	B50 CEM III
CEM I	37.5%	20.0%	50.0%			
CEM III				70.0%	60.0%	50.0%
Silica fume	32.5%	32.5%	50.0%	30.0%	40.0%	50.0%
Fly ash	30.0%					
Slag		47.5%				
SiO_2 content	≈ 54%	≈ 56%	≈ 60%	≈ 50%	≈ 57%	≈ 64%

Nine commercial CEM I as well as three commercial CEM III (produced in France) were selected. Most of them were "local" products (cements produced as close as possible to Saint-Dizier, where the FSS test box was built). The commercial silica fume Condensil® S95-DM was selected because it was already used in previous studies and turned out to be more efficiently dispersed than other known products. It must be noted though that silica fume particles inevitably tend to flocculate thus partly impairing the silica dissolution.

The binder composition effect on the pH decrease was studied on constantly stirred aqueous suspensions. A small amount of binder (2.5 g) was diluted in a bigger amount of deionized water (25.0 g). Siliceous sand (crystallized quartz, 20.0 g, diameter 3-4 mm) was also added to prevent hardening and to accelerate hydration (via reactive surface grinding). The mixtures were prepared in 50 mL centrifugation tubes hermetically sealed (Figure 5). They were kept in constant agitation (using a tube rotator, 50 rpm) in a CO^2 free glove box, in an air-conditioned room (20 ± 2°C). At regular intervals, the rotator was stopped and the aqueous suspensions were left to settle a little to measure the pH value of the solution at equilibrium with the solids. About fifty aqueous suspensions with different binder compositions were tested.

View of the 50 mL tube (with the sealing cap) containing the binder aqueous suspension

View of the tube rotator

Figure 5: Measure of aqueous suspensions pH as a function of the binder composition.

The evolution of the pH value obtained for the different binder compositions is shown in Figure 6. The top horizontal line (pH=12.6) represents the value at equilibrium with portlandite at 20°C (without alkalis) whereas the colored zone between pH=10.5 and pH=11.0 represents the FSS target zone, after 28 days. Whatever the binder, the pattern was quite the same: in a first time the pH value remained almost constant (12.6, a value due to the presence of portlandite) and then constantly decreased. It was noticed that for a given binder composition, the origin of the cement (and the alkali content) was not found to have a significant influence on the pH decrease and ultimate asymptotic value.

The decrease rate was found to depend on the binder composition. The two ternary mixes yielded slow pH decrease; after 90 days and whatever the considered composition (T1 and T3) the pH remained greater than the specified value (11.0), while the blend including slag (T3) proved to give the faster pH decrease of the two.

The fastest pH decrease of all binder compositions was obtained by using the B50 CEM I mix with a pH value lower than11.0, reached after only 35-40 days. The results of the B50 CEM III showed the significant influence of added silica: the higher the silica fume content, the faster the pH decrease. Silica fume incorporation up to 30% gave similar results in the ternary mixes, whereas 40% gave similar results to the B50 CEM I mix. The use of a 50% incorporation enabled being very close to the pH requirement.

The pH values reached after 28 days by the two ternary mixes were far above the specified value. These two binder compositions were then judged inadequate and rejected. The B50 CEM I as well as the B40 CEM III mixes which gave interesting results (though not fully satisfactory) were kept. The B50 CEM III mix, which yielded the best pH results, was also selected. To summarize, the three following binder compositions were selected for the next phase (concrete proportioning in the laboratory):

- B50 CEM I (50% CEM I + 50% silica fume),
- B50 CEM III (50% CEM III + 50% silica fume),
- B40 CEM III (60% CEM III + 40% silica fume).

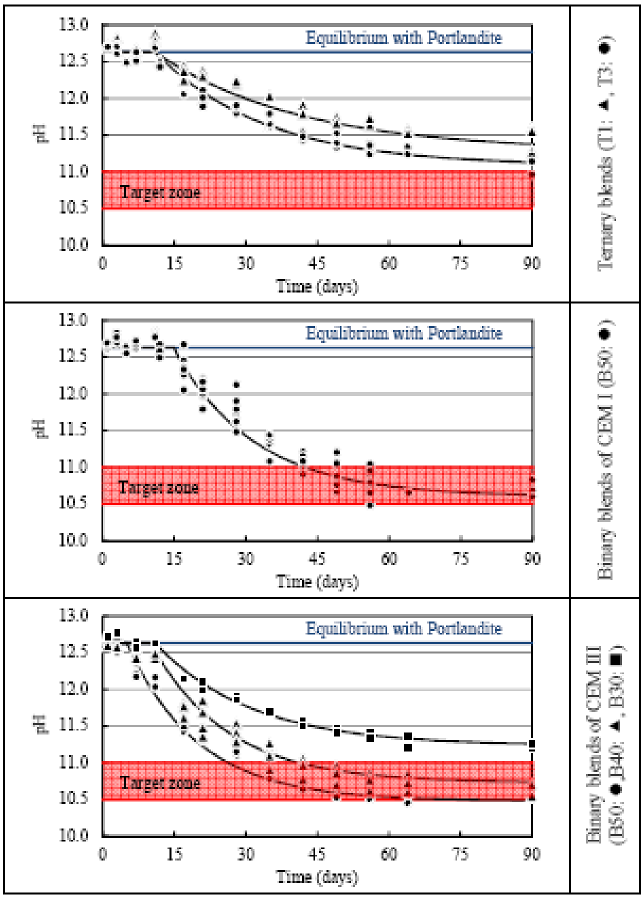


Figure 6: pH variations vs time of the aqueous suspension as a function of the binders.

4.3 Concrete mixture proportioning

Concrete mixture proportioning was studied by CEA-LECBA using the software "Betonlab Pro 3" which easily allows estimating the properties of any concrete mixture from the fresh state to the hardened state.

The main drawback of such an approach is the large number of parameters needed for the full description of the raw materials and the resulting concrete properties. Not all of them could be acquired before the proportioning study; it was then decided to focus on the fresh state rheology and the compressive strength.

Thus concrete mixture proportioning was performed using an iterative approach (Figure 7):

- In a first step, the initial FSS requirements were transposed into requirements that could be managed by the software and the concrete mixes were designed.
- The second step consisted in producing the concrete mixes to check their feasibility and verify their rheology.
- If satisfactory, the other properties (pH of the pore solution, mechanical strength) were also measured.

All those operations were constitutive of the so called iteration. Any significant difference between the resulting concrete properties and the expected ones involved changes in the requirements and the beginning of a new iteration.

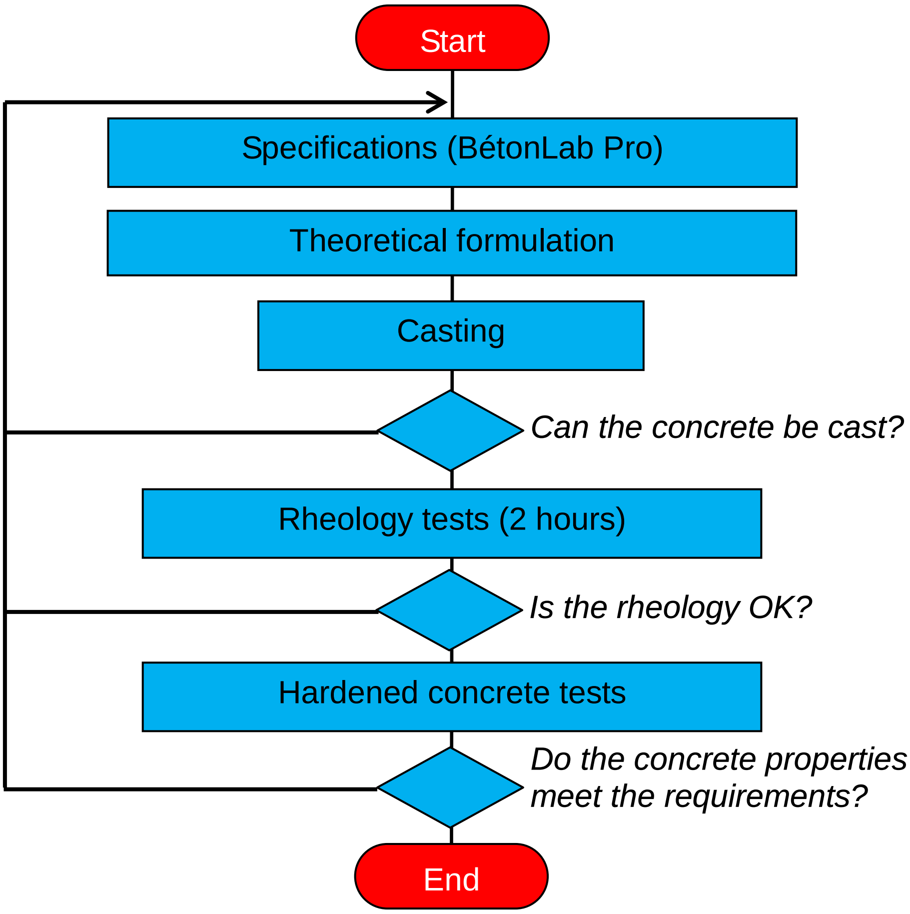


Figure 7: Iterative approach used for LA-SCC proportioning.

The initial requirements were translated into the following specifications in the Betonlab application and for the first iteration loop:

- The compressive strength at 28 days must be greater than 40 MPa.
- The yield stress and the viscosity of the fresh material must be lower than 400 Pa and 200 Pa/s respectively (these values were specified to ensure self-compaction).
- The initial bleeding rate must be lower than 0.4×10^{-5} m/s to avoid bleeding and blockages in the pumping process.
- There must be no discontinuity in the granular skeleton to prevent segregation and ensure pumpability.
- The compaction indices of the big aggregates (5/12.5 mm) Kgg and fine particles (diameter lower than 80 µm) Kf must be lower than 1.4 and greater than 3.5 respectively.
- The aggregates to sand ratio must be ranging between 0.8 and 1.2 to limit the amount of aggregates according to current recommendations.

Among all the binder compositions tested in the first phase, the four ones that yielded the fastest pH decrease were selected for the LA-SCC production phase, namely:

- B50 CEM I 52.5 N PM-ES-CP2 Le Teil (Lafarge);
- B50 CEM III/A 52.5 L PM-ES-CP1 Rombas (Calcia);
- B50 CEM III/A 42.5 N LH PM-ES Héming (Holcim);
- B40 CEM III/C 32.5 N PM-ES Rombas (Calcia).

Local calcareous aggregates were supplied: rounded gravel 5/12.5 mm and crushed sand 0/4 mm. The commercial limestone filler Carmeuse Premiacal® was used to improve the segregation resistance of the fresh mix. The superplasticizer BASF Glenium® Sky 537 was used to achieve the targeted level of fluidity and the retarding admixture BASF Prelom® 510 was used to maintain the rheology for two hours after mixing.
Alternatively, the superplasticizer could have been used alone either by adjusting the initial dosage so that the slump flow was greater than 650mm two hours after mixing or by a new addition of superplasticizer before use (two hours after mixing). However, the use of the retarding admixture was preferred because this methodology offers the great advantage to counterbalance the effect of ambient temperature by adjusting the retarding additive dosage (the LA-SCC was planned to be concreted day and night in summer time).

4.4 Concrete tests in lab

Once designed, the concrete mixtures were tested in the CEA-LECBA laboratory: a 30 L batch of each mix was produced using an 80 L planetary mixer (Figure 8). Before each batch, the aggregates (sand and gravel) moisture content was measured and the amount of added water was adjusted (reduced) to compensate the water brought in by the aggregates. All the dry compounds (sand, gravel, cement, filler and SCMs) were stirred together for two minutes before the water together with the additives were added. The mixture was then mixed for an additional five minutes period. The total mixing time was longer than usual but it was needed to ensure satisfactory homogeneity.

Figure 8: Concrete preparation in lab mixer.

The slump flow test (Figure 9) was used to test “flowability”. In addition to the spread diameter measure, some visual examination also gave valuable information on the concrete quality (distribution of aggregates, aggregate/paste separation, ring of water). The segregation resistance was tested using the sieve segregation resistance test. The temperature increase under semi-adiabatic conditions due to the heat of hydration was measured following the European standard EN 196-9 (Langavant calorimeter). The compressive strength was measured after 28 and 90 days following the European standard EN 12390-3 using each time 3 cylindrical specimens (Ø11×22 cm) cured in water. Porosity was measured using cores (Ø4×6 cm) taken out of bigger specimens (Ø11×22 cm). The pH of the pore solution was assessed using small specimens (Ø6×10 cm) kept in sealed containers. Some 28 and 90 days after casting, a high-pressure extraction device was used to express the concrete pore solution, the pH of which was measured in a CO^2 free glove box.

Figure 9: Slump test measurement.

From a practical point of view, all the concrete mixtures designed using “Betonlab Pro” could be produced for real: suitable additive dosage could be found to achieve the required properties at the fresh state (slump flow and segregation resistance after two hours). It must be noted, though, that the resulting SCC mixes were quite unusual (aspect): the cement content was low whereas the filler was very high. The SP dosage was also very high, in relation to the high amount of silica fume.

Only two iterations were necessary in the proportioning process to obtain the target properties. The compressive strength of the first iteration concrete formulations was found to be too high (about 25-30 MPa after 7 days). This was attributed to the aggregates influence that could not be evaluated before the concrete mixture proportioning phase.

The targeted compressive strength after 28 days was then reduced by 15 MPa in the second iteration (Table 2). As a result, the compressive strength values measured after 28 days ranged between 37 MPa and 48 MPa; that is to say some 7 MPa to 18 MPa over the specified value (Table 3). This difference could have been reduced by lowering the targeted strength in a third iteration, but it was kept as it was to counteract the potential strength decrease commonly observed on concretes used in real in situ conditions.

Table 2: LA-SCC mix proportions lab tested in the 2nd iteration. The figures in brackets represent the additive as a percentage of binder mass (cement + silica fume + filler).

Compound (kg/m³)	B50 CEM I	B50 CEM III/A		B40 CEM III/C
	52.5 Le Teil	52.5 Rombas	42.5 Héming	32.5 Rombas
Gravel 5/12.5 (dry)	807.7	682.1	737.8	838.3
Sand 0/4 (dry)	762.1	698.7	656.6	766.0
Cement	108.0	130.0	132.0	147.0
Silica fume	108.0	130.0	132.0	98.0
Filler	335.5	408.4	396.9	278.8
Water	178.2	204.1	205.7	174.2
Glenium 537	16.545 (3.0%)	14.705 (2.2%)	13.218 (2.0%)	14.666 (2.8%)
Prelom 510	1.655 (0.3%)	2.005 (0.3%)	1.983 (0.3%)	1.571 (0.3%)

The porosity values were found to be high (between 16% and 19%) in relation with the high amount of silica fume incorporated (the B40 CEM III/C with the lowest silica fume content presented the lowest porosity value).

The pH of the pore solution was greater than the required value after 28 days (11.0). The measured values (cf. Figure 10: expression of the pore solution) were similar to the ones acquired using the aqueous suspensions.

The values measured after 90 days were however lower than 11.0 (between 10.3 and 10.8), probably at equilibrium (or at least very close to).

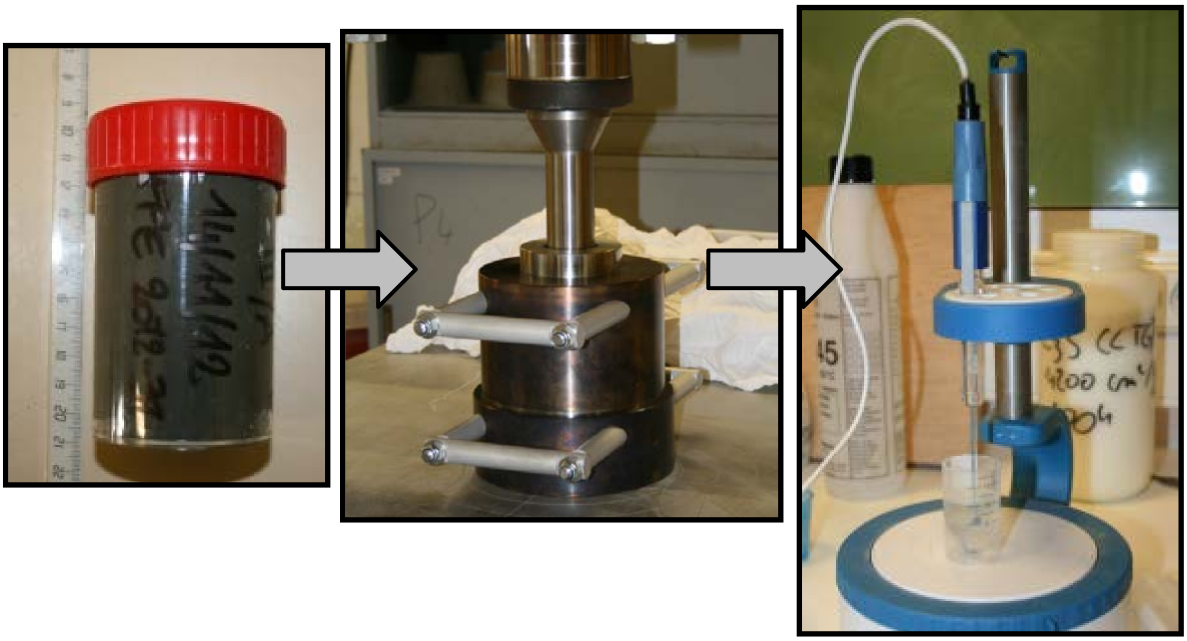


Figure 10: Expression of the pore solution.

The maximal temperature increase under semi-adiabatic conditions was found to be approximately 4°C, whatever the mix. This was a very small temperature increase value compared to usual concretes and even to low-heat ones (Figure 11).

Table 3: Main properties of the LA-SCC mixes

Concrete mix	Strength (MPa)		Specific gravity	Porosity to water	pH	
	28 d	90 d	28 d	28 d	28 d	90 d
B50 CEM I 52.5 Le Teil	47.7	59.5	2.33	17.1%	12.0	10.5
B50 CEM III/A 52.5 Rombas	41.8	50.1	2.31	18.8%	11.3	10.4
B50 CEM III/A 42.5 Héming	43.2	54.6	2.30	18.4%	11.5	10.3
B40 CEM III/C 32.5 Rombas	37.1	46.8	2.34	16.1%	11.8	10.8

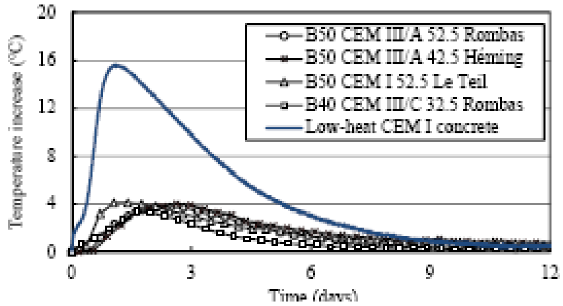


Figure 11: Temperature increase of the LA-SCC under semi-adiabatic conditions and comparison with a low-heat CEM I concrete.

4.5 Testing of SCC concretes at a metric scale in batches (in field)

First, the LA-SCCs were produced using the concrete plant (i.e. the "Paul Calin" concrete mixing plant, in Saint-Dizier, some 5 km from the FSS construction hangar, cf. Figure 12) selected by the FSS project, in order to verify the feasibility in industrial conditions of the mixes developed in the laboratory; i.e. check and adjust the additives content and adapt the production process.

In so doing only the additives dosages were slightly modified; all the other parameters were left unchanged. The concrete plant was equipped with a 2 m^3 planetary mixer and was typical of concrete plants operated in France (Figure 12). The staff operating the plant had no anterior experience of silica fume use or of concretes with large additions.

The LA-SCCs were produced using 500 L batches following the mixing protocol used in the laboratory. Immediately after mixing the LA-SCCs were discharged in a truck mixer and the slump flow and segregation resistance parameters were monitored over a two hours period (about 75 liters of concrete were poured in a wheelbarrow for each sampling). These tests were implemented outside and in winter time: the concrete temperature was much lower than in the LECBA laboratory (Table 4). It was possible to produce all the LA-SCC mixes but not without endeavor. Table 4 illustrates the difficulties encountered and the temperature effect on the retarding additive content.

The first batch was realized using the additives dosage adjusted in the laboratory (2.2% Glenium Sky 537 and 0.3% Prelom 510). The result was not satisfactory: the slump flow was lower than 650 mm. Increasing the superplasticizer content (batches #2 and #3) did not improve the result: the fresh mix was unstable. Finally, reducing the retarding admixture dosage from 0.3% to 0.1% (batches #4 and #5) proved to be the most suitable solution due to the low temperature. The final batch (#5) gave satisfactory results: the slump flow and segregation resistance were adequate just after mixing and also two hours later. It must be noted that in this case the superplasticizer dosage was the same as in the laboratory. These results emphasized the strong influence of temperature on the fresh mix rheology and the need to adjust the additive content to effective ambient temperature.

Figure 12: Concrete mixing plant & truck mixer (left) /sampling from the truck mixer (right)

Table 4: Industrial-scale batching of the B50 CEM III/A Rombas 52.5 concrete mix (SP: superplasticizer Glenium Sky 537, RA: retarding additive Prelom 510).

Batch	Temperature		Admixtures	Slump flow		Sieve test	
	Air	Concrete		t_0	t_0+2h	t_0	t_0+2h
#1	1°C	6°C	SP 2.2% + RA 0.3%	550	500	-	-
#2	1°C	5°C	SP 2.7% + RA 0.3%	750	840	40%	-
#3	2°C	6°C	SP 2.45% + RA 0.3%	730	850	22%	-
#4	4°C	9°C	SP 2.4% + RA 0.1%	710	730	13%	25%
#5	4°C	9°C	SP 2.2% + RA 0.1%	660	680	11%	11%

In a second step, three "test cubes" (1×1×1 m^3) were fabricated to validate the methodology chosen for the construction of the LA-SCC containment wall at full scale and compare the concrete properties, in order to select the final formulation. The concretes were produced by the same concrete plant in two consecutive batches (2×1.5 m^3). When the first batch was ready, the concrete was poured into a 7 m3 truck mixer and the second batch was prepared and eventually poured into the same truck mixer (the mixing operations for each batch took approximately 20 minutes). The truck mixer was then driven to the FSS mockup construction site (this operation taking about 10 minutes) where the concrete was kept in the (slowly revolving) truck mixer in the waiting. Two hours after pouring the second batch, the concrete was pumped over 10 meters into the mold (formwork) and left without vibration or levelling (Figure 13). Samples were fabricated for further verification of the compressive strength and others were cored out of the cubes to measure the concrete properties (compressive strength, porosity and pH) in real in situ conditions.

Figure 13: Truck mixer and pumping boom (left) & pouring inside metric cubic mold (right).

The three tested LA-SCC mixes were the following (Figure 14):

- B50 CEM I 52.5 Le Teil;
- B50 CEM III/A 52.5 Rombas;
- B50 CEM III/A 42.5 Héming.

Figure 14: The three cubic metric scale batches.

Due to the low ambient temperature, the retarding additive dosage for the CEM III/A mixes was set at 0.1% of the binder weight (Table 5). The B50 CEM III/A 52.5 Rombas could be pumped without difficulties and showed acceptable slump flow and segregation resistance for two hours. The superplasticizer content of the B50 CEM III/A 42.5 Héming was increased to 2.2% to anticipate insufficient fluidity (following the results of the feasibility tests), but this proved not to be necessary. The slump flow value was higher than expected but the concrete remained pumpable, self-compacting and stable despite high fluidity. The B50 CEM I 52.5 Le Teil cube was elaborated later at a higher ambient temperature. The retarding admixture dosage was then increased to 0.2%. Here again the concrete could be pumped without any difficulty and the fresh mix showed suitable behavior.

The measured properties at 28 days (Table 6) were somewhat different from those obtained in the laboratory (lower strength and higher pH and porosity). It was believed that the decrease in temperature (the onsite temperature was lower than in the LECBA laboratory) had slowed down hydration. This assumption was supported by measuring the strength difference between the samples cored from the cast cubes (kept onsite) and the specimens (later stored in the laboratory).

Table 5: Properties of the different concretes tested, at the fresh state

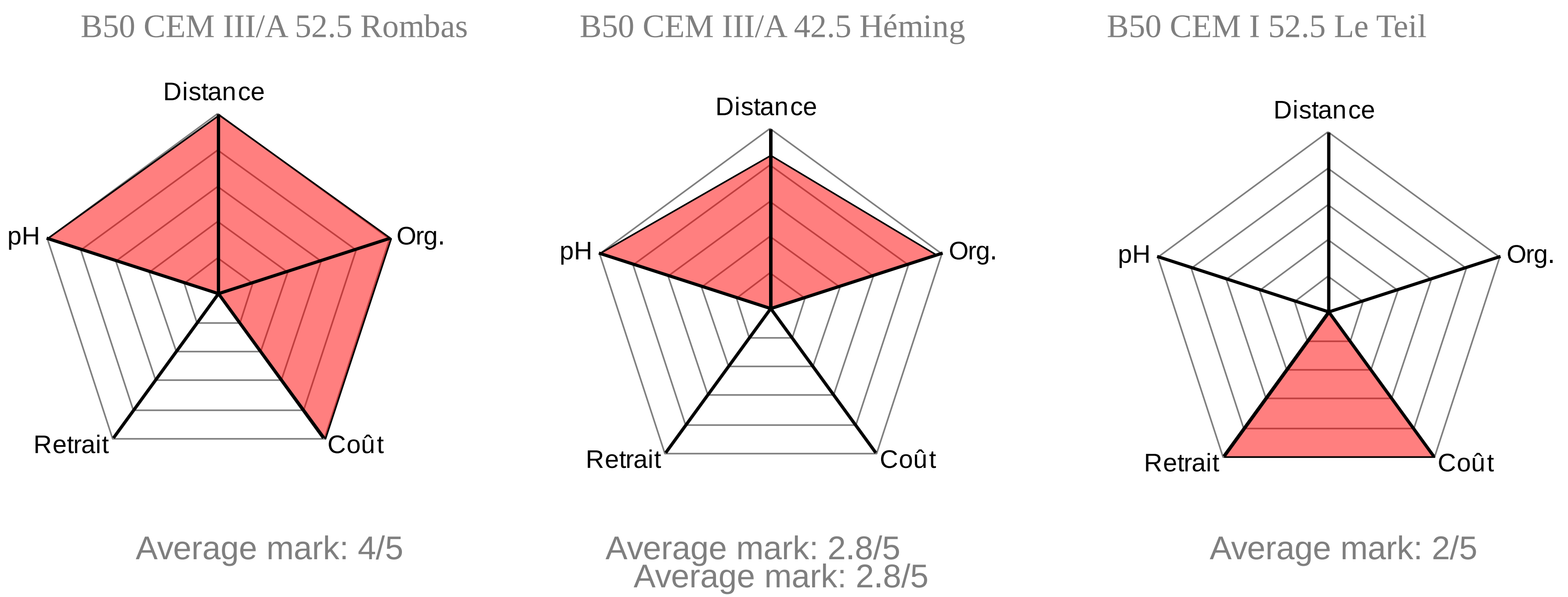
Properties		B50 CEM I 52.5 Le Teil	B50 CEM III/A 52.5 Rombas	B50 CEM III/A 42.5 Héming
Date of test		2013/03/13 am	2013/03/13 pm	2013/04/04 pm
Temperature	Air	2°C	3°C	9°C
	Concrete	10°C	9°C	16°C
Admixture	Glenium	2.2%	2.2%	3.0%
	Prelom	0.1%	0.1%	0.2%
Slump flow (mm)	t_0	650	770	700
	t_0+2h	660	740	720
	t_0+2h	640	720	660
Segregation resistance	t_0	*25%**	*13%**	16%
	t_0+1h	-	-	14%
	t_0+2h	15%	18%	7%
Pumpability		OK	OK	OK
Remarks		-	Too fluid	-

** Dubious value*

Table 6: Properties of the different concretes tested, at the hardened state

Concrete mix	Compressive strength (MPa) Standard specimens		Compressive strength (MPa) Cores	Specific gravity	Porosity	pH	
	28 d	90 d	28 d	28 d	28 d	28 d	90 d
B50 CEM I 52.5 Le Teil	46.7	61.9	46.2	2.27	17,8%	12.2	10.3
B50 CEM III/A 52.5 Rombas	37.8	50.7	34.9	2.22	19,7%	11.8	10.1
B50 CEM III/A 42.5 Héming	36.9	49.6	31.3	2.21	19,9%	11.8	10.2

Comparison of these operational results crossed with the cost of the 3 admixtures led to the choice of B50 CEM III/A 52.5 Rombas as the concrete mix most suited for the future construction of the LA-SCC containment wall. This selection method (a multi-criteria analysis) is illustrated in Figure 15.



⇒ Selection of the mix B50 CEM III/A 52.5 Rombas

Figure 15: Multi-criteria analysis of the reference concrete formulations for selection of the best recipe.

Figure 16 presents the pH decrease of the selected mix (B50 CEM III/A 52.5 Rombas) measured using the high-pressure pore-solution extraction from samples cast and kept under autogenous conditions in the LECBA laboratory. Unlike the aqueous suspensions used to study the binders, the initial value was higher than 12.6 (in fact 12.9) due to reduced alkalis dilution. After five days the pH value sharply decreased to become lower than 11.0 after about 30 days and reached 10.5 after 90 days. It is noteworthy that the pH results obtained using concrete samples and aqueous suspensions showed a very similar evolution versus time.

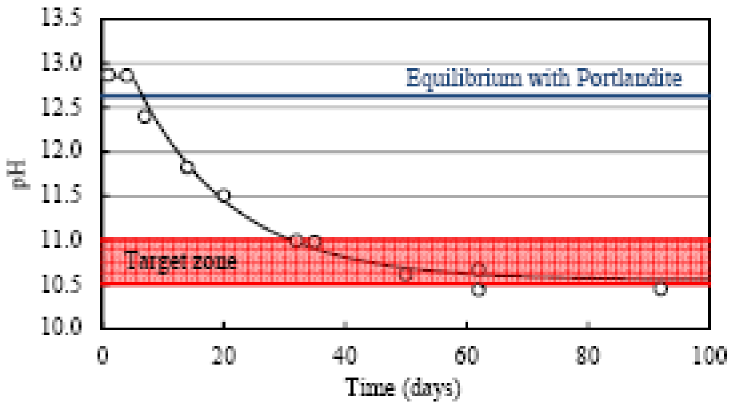


Figure 16: Evolution of the pH of the pore solution of the B50 CEM III/A 52.5 Rombas concrete mix as prepared in the LECBA laboratory

5. Conclusion

The feasibility of the selected LA-SCC production in industrial conditions was validated by the metric scale tests carried-out in field.

It was beheld that 5 minutes were far more than necessary for the wet mix to be homogenized. It was then proposed to reduce the mixing time to 100 seconds after introduction of water (and additives) in order to boost the LA-SCC production rate. Following the current guidelines (EFNARC, 2005) it was also recommended to modify the acceptance criterion of the ready-mixed concrete pertaining to slump flow: ± 80 mm of target value (570 mm ≤ slump flow value ≤730 mm). The same was suggested for the segregation resistance test: SR ≤ 23% (instead of 20%).

The FSS containment wall construction being scheduled mid-July 2013, temperature was found to have a big impact on the LA-SCC behavior at the fresh state. It was then suggested to keep untouched the SP dosage and to adjust the RA content to the effective temperature: the higher the temperature, the higher the RA dosage.

Moreover it was required that the maximal temperature reached should be less than 50°C. Using the results from the semi adiabatic calorimetry, the heat emitted during hydration was assessed (following the standard EN 196-9) and used to estimate the maximal temperature at which the concrete could be used to meet the requirement (in doing so, all the heat emitted was assumed to participate in the temperature increase). It was found that the maximal temperature increase was 24°C. It was then advised not to pour concrete with a temperature greater than 26°C.

The final low pH SCC recipe used for the casting of the FSS upstream containment wall is given in Table 7 (it includes a fine-tuning of the additives to account for temperature).

Table 7: Composition of the concrete mixture as used in FSS for the SCC containment wall construction.

Components	Quantity
Rounded Gravel 5/12 (dry)	682.1 kg/m3
Sand 0/4 (dry)	698.7 kg/m3
Cement ROMBAS CEM III/A	130.0 kg/m3
Silica Fumes	130.0 kg/m3
Filler	408.4 kg/m3
Glenium SKY 537 (SP)	2.2%
Prelom 510 (RA)	0.1%
Water	204.1 kg/m3

The construction of the upstream low pH self-compacting concrete containment wall, which started in mid-July 2013, is detailed in the DOPAS Deliverable D3.11 “Report on FSS cast concrete plug construction”.

6. References

- Andra 2005 - Dossier 2005 Argile: “Safety evaluation of a geological repository”.
- Andra 2012 - Cahier des charges FSS1: “Etude et réalisation du démonstrateur technologique de scellement à pleine échelle (Full Scale Seal)” - ref. CG.TE.F.CDC.AMOA.GC0.2000.12.0014.
- DOPAS Deliverable D2.1 “Design Bases and Criteria”.
- DOPAS Deliverable D3.2 “FSS Tunnel model design report”.
- DOPAS Deliverable D3.10 “FSS drift model construction report”.
- DOPAS Deliverable D3.11 “Report on FSS cast concrete plug construction”.