

Cebama reference mix design for low-pH concrete and paste, preliminary characterisation

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Abstract

A reference low-pH concrete and paste mix were manufactured within the Cebama-project. Reference mixtures were casted at VTT in March 2016 and were distributed among some of the Cebama partners. Reference mix designs will be used by different partners as a common material to study their behaviour in contact with waters of different composition and interaction with radionuclides. Additionally, these materials will be used for model calibration. This article provides a summary of the characterization methods used by different partners and gives an overview of the experiments which will be made in the future with the reference materials.

Introduction

A reference low-pH concrete and paste mixture for the Cebama-project was manufactured in March 2016 by VTT and distributed to some of the partners. Reference mix designs enable comparison of various research methods applied in the Cebama-project. Mix designs of the reference concrete (RCM) and reference paste (RPM) are presented at Table 1. The basis of the Cebama reference concrete was the low-pH concretes developed in the FP7 Dopas-project (Holt and Koho, 2016). The composition of the Cebama RCM and low-pH concretes have identical mix design of fillers and coarser aggregates. Also the CaO/SiO₂ -ratio of the mix designs is identical. The only factor that differ is the binder composition. The Cebama RCM has a ternary binder composition (cement 105 kg/m³, silica fume 110 kg/m³ and blast furnace slag 65 kg/m³), whereas the ternary low-pH concrete from the

Dopas-project has a cement 105 kg/m³, silica fume 91 kg/m³ and fly ash 84 kg/m³. The binder composition of binary low-pH concrete from the Dopas -project consisted of cement 120 kg/m³ and silica fume 80 kg/m³. More detailed information of the sample castings and mix design is described in the 1st Annual Workshop Proceedings of Cebama-project (Vehmas et al., 2016).

This contribution provides an overview of the preliminary results performed to characterize the RCM and RPM. The article presents preliminary results from the partners and summarizes the upcoming studies.

The data from the reference mixture assessments will be supplied for WP3 - modelling and various parameters can be extracted from the data. The studies also provide common references in methodologies which allow better comparison to locally characterized materials by different partners.

Table 1: Mix designs of Cebama reference concrete- and paste-mix designs.

Materials	Concrete (RCM)	Paste (RPM)
CEM I 42.5	105 kg/m ³	468 kg/m ³
Silica fume	110 kg/m ³	491 kg/m ³
Blast furnace slag	65 kg/m ³	290 kg/m ³
Quartz filler	116 kg/m ³	517 kg/m ³
0 - 1 mm	168 kg/m ³	-
0 - 8 mm	770 kg/m ³	-
8 - 16 mm	532 kg/m ³	-
16 - 32 mm	396 kg/m ³	-
Water (effective)	120 kg/m ³	312 kg/m ³
Superplasticizer*	16.80 kg/m ³	75 kg/m ³
Water / binder -ratio	0.43	0.25

*Superplasticizer was naphthalene sulfonate -based Pantarhit LK FM from HaBe.

Research methods

Cebama reference concrete (RCM) and paste (RPM) were analysed with multiple methods (Table 2). Authors' contributions have been labelled with the superscript corresponding to the numbers described in the address lines. Besides the authors, University of Sheffield (USFD), Autonomic University of Madrid (UAM) and The French Geological Survey (BRGM) are performing additional studies related to the reference mix designs. The French Geological Survey results of spectral induced polarization (SIP) for reference -concrete (RCM) and -paste (RPM) are presented in a separate article (Huisman et al., 2017).

Table 2: Cebama reference concrete and paste characterization methods.

Quality	Analyzer	Paste (RPM)	Concrete (RCM)
<i>Fresh-stage properties</i>			
Workability	VTT ¹	yes	yes
Air Content	VTT ¹	no	yes
Heat of hydration	USFD	yes	no
Setting	USFD	yes	no

Quality	Analyzer	Paste (RPM)	Concrete (RCM)
<i>Mechanical properties</i>			
Compression strength	VTT ¹ , USFD, CTU ⁵	yes	yes
<i>Chemical composition</i>			
X-ray diffraction (XRD)	KIT ⁸ , USFD, JUELICH ⁷ , SURREY ³ , CSIC ² , UAM	yes	yes
X-ray fluorescence (XRF)	SURREY ³		initial materials
Thermogravimetry (TG/DTA)	KIT ⁸ , USFD, CSIC ² , UAM	yes	yes
²⁹ Si and ²⁷ Al MAS NMR	KIT ⁸	yes	no
Energy dispersive microscopy (SEM, Back Scattering+ EDS)	KIT ⁸ , CSIC ² , USFD, JUELICH ⁷	yes	yes
Pore solution pH	KIT ⁸ , VTT ¹ , CSIC ²	yes	yes
<i>Microstructure</i>			
Scanning electron microscopy (SEM)	KIT ⁸ , CSIC ² , USFD, JUELICH ⁷	yes	yes
Porosity	CSIC ² , USFD, UJV ⁶	yes	yes
X-ray computed tomography	USFD	yes	no
<i>Transport properties</i>			
Leaching	VTT ¹	yes	yes
Percolation	USFD, SURREY ³ , CSIC ² , CTU ⁵ , UJV ⁶	yes	yes
Diffusion	JUELICH ⁷ , CSIC ² , UAM, KIT ⁸	yes	yes
<i>Other</i>			
Density	VTT ¹ , USFD	yes	yes
Spectral induced polarization (SIP)	JUELICH ⁷ and BRGM	yes	no

Results

Results from the reference concrete and paste mix designs from Cebama partners are presented in the next chapters. The results are divided into concrete and paste studies as their properties are not identical. During the castings of the reference mix designs, it was decided to obtain the excellent mechanical properties of reference concrete to the reference paste. As a consequence, the chemical properties of the concrete and paste can differ due to the lower total water content used in the Cebama reference paste mix design.

Reference concrete mix design (RCM) studies

Studies related to the characterization of the RCM have been mainly performed by CSIC and VTT and are briefly described below.

CSIC

All tests carrying out by CSIC relate to the concrete reference mix (RCM). Tests refer to:

1. Identification of chemical / microstructure evolution during hydration by XRD, DTA/TG, BSEM + EDS, MIP (mercury intrusion porosimetry) and pH measurement.

2. Characterisation of alteration transport properties through ground water percolation column test (with Granite Grimsel water and simulated clayey water) and natural Cl diffusion test.

In addition to RCM studies, CSIC is studying a similar concrete mix dosage (in cement + supplementary cementitious materials + aggregates content) to the RCM but higher w/b ratio = 0.6 (air content = 2.7% and low density = 2,320 kg/m³). This new concrete will be identified as RCM-LD (Low Density). The aim of having this new concrete is to favour the transport properties that allow to identify changes in processes during the Cebama project. Characterisation of the initial materials, percolation and Cl diffusion tests will also be carried out in this RCM-LD concrete.

The tests conditions and progress are detailed in Table 3.

Table 3: Test conditions and progress of the initial (Init) characterization of the RCM and RCM-LD.

Concrete type	Init. pH (Alonso et al., 2012)	Init. water soluble pore Ion	Init XRD	Init DTA/TG	Init BSEM+EDS	Init MIP
RCM*	yes	yes	yes	yes	yes	yes
RCM-LD**	yes	In progress	yes	In progress	In progress	In progress

*age from casting: 5 months, **age from casting: 2 months

The pH in both concretes is below that from a Ca(OH)₂ saturated solution (12.5), values measured are: RCM (11.47 ± 0.03) and RCM-LD (11.57 ± 0.06). XRD tests were carried out of enriched powder samples of cement paste removing the coarse aggregates. XRD results do not reveal the presence of portlandite. Sulfoaluminate phases, as ettringite, are also not evident at this age of hydration. The calcium-silicate-hydrate (C-S-H) crystalline phases are not clearly identified due to the interference with aggregates. The absence of portlandite is also confirmed by TG/DTA. The BSEM images in RCM show a dense microstructure and good aggregate-cement paste of interfacial transition zone (ITZ). The EDS reveal a mean CaO/SiO₂ ratio = 0.55 ± 0.17 and a CaO/Al₂O₃ ratio = 8 ± 4.6. MIP confirms the low total porosity in RCM (2.3%) with pores distributed below 0.02 µm, (mean pore size 0.01 µm), all this agree with the high density of this concrete (2.42 g/mL).

Table 4 summarizes the tests carried out to characterise the evolution of transport properties in RCM and RCM-LD. The first results with RCM, during 100 days of operation in percolation tests at 8 bars show high resistance to percolation of water due to its low porosity. The hydraulic conductivity from the few ml of water percolated with time gives values between 10⁻¹³ to 10⁻¹⁴ m/s. The pH of the first eluted water shows values of 8.1 with clayey water.

Table 4: Transport properties characterisation tests.

Concrete type	Percolation		Cl natural Difussion
	P (bar)	3 alteration ages	At 12, 24 months
RCM	8	In progress (100 d)	Future
RCM-LD	1	In progress (7 d)	Future

VTT

VTT has measured the fresh-stage properties of the reference concrete (RCM). Slump of the fresh reference concrete was 180 mm. Density of the fresh concrete was 2,450 kg/m³ with air content 0.9% (SFS-EN 12350-7,

2009). Compression strengths and densities of the reference concrete at various ages are presented in Table 5. Compression strength results and densities of Dopas-project binary and ternary low-pH concretes are also presented in Table 5 for comparison.

Table 5: Compression strengths and densities of RCM, Binary- and Ternary mixtures developed in the Dopas-project.

Age	Compression strength	Density
<i>Cebama Reference Concrete (RCM)</i>		
7 d	47.0 MPa	2,420 kg/m ³
28 d	80.0 MPa	2,430 kg/m ³
424 d	88.5 MPa	2,420 kg/m ³
<i>Binary low-pH Concrete</i>		
91 d	95.5 MPa	2,420 kg/m ³
483 d	112.5 MPa	2,420 kg/m ³
<i>Ternary low-pH Concrete</i>		
91 d	80.0 MPa	2,400 kg/m ³
475 d	100.0 MPa	2,420 kg/m ³

pH-development of RCM was determined according to the method described in the literature (Alonso et al., 2012). Results are presented in Table 6. The results are accompanied with pH-values of Binary- and Ternary low-pH concretes.

Table 6: Measured pH-values of RCM, along with Binary- and Ternary- mixtures developed in the Dopas-project.

Age	pH
<i>Cebama Reference Concrete (RCM)</i>	
8 d	12.16
19 d	11.94
28 d	11.92
428 d	11.47
<i>Binary low-pH Concrete</i>	
59 d	11.65
91 d	11.55
486 d	11.29
<i>Ternary low-pH Concrete</i>	
59 d	11.59
91 d	11.59
486 d	11.29

Reference paste mix design (RPM) studies

Studies related to the characterization of the RCM concrete have been mainly performed by KIT, SURREY, CTU, UJV, JULICH, and VTT and are described briefly below.

KIT

KIT-INE has mainly focused on the chemical and microstructure characterization of the cement paste using X-ray diffraction (XRD), thermogravimetric - differential thermal analysis (TG-DTA), ²⁹Si and ²⁷Al magic angle spinning nuclear magnetic resonance (²⁹Si and ²⁷Al MAS NMR) and scanning electron microscopy - energy dispersive X-ray spectroscopy (SEM-EDX). The analysis of the pH of the solid was also measured using the *ex-situ* leaching method described by Alonso et al. (2012).

The analysis of the pH of pore water results in a value around 11.7. The X-Ray diffractogram of the paste identified quartz as the main crystalline phase present in the solid. C-S-H phases could not be detected in any of the samples probably due to its amorphous nature. Furthermore, ettringite (Ca₆Al₂(SO₄)₃(OH)₁₂·26H₂O) has been identified as a minor phase. TG- DTA shows a loss of mass around 100 - 300°C which is attributed to C-S-H phases and ettringite. However, in this temperature range the mass loss can also be attributed to water bound to mineral surfaces (10.9%) making a quantification of the of C-S-H phases difficult. The weight loss at 450°C which is indicative for portlandite is not observed confirming the absence of this solid phase.

The pastes were also analyzed by SEM-EDX to observe the morphology and chemical composition of the various hydrated phases. C-S-H phases, non-reacted silica fume (with a characteristic spherical shape), quartz and feldspar are detected. Additionally, Ca/Si-ratio of the C-S-H phases are determined which is presently not possible by the other techniques used in this study. SEM-EDX revealed low Ca/Si-ratios between 0.5 and 0.7. To complete the interpretation of the data and to show the homogeneity of the sample at the microscale range, elemental maps of silicon, calcium, iron, magnesium, aluminum and sulphate have been produced (Figure 1). The various mineral phases present are characterized by the local concentrations of calcium, silicon and iron.

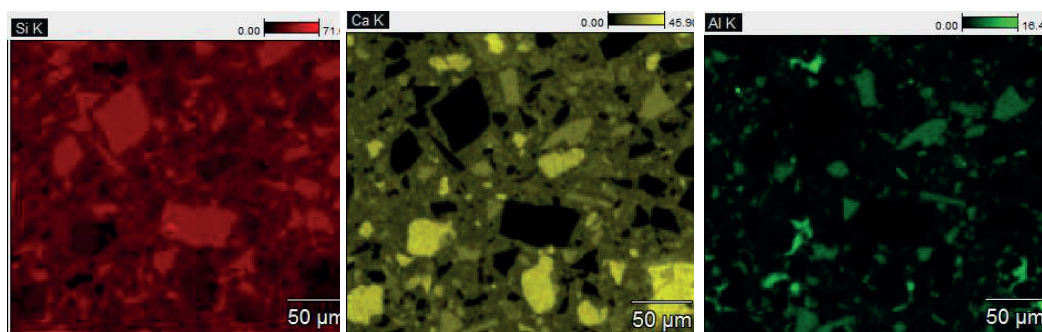


Figure 1: SEM-EDX elemental maps of silicon (red), calcium (yellow), and aluminum (green).

Solid state NMR-spectroscopy was applied to the samples. The main advantage of solid state NMR spectroscopy is its ability to detect nuclei in amorphous phases and its possibility to provide structural information. The ²⁹Si MAS NMR spectra is depicted in Figure 2. A broad signal in the chemical shift range between -75 and -100 ppm is the main signal of the spectrum and can be assigned to the Si present in the C-S-H phases overlapped with the signal of feldspar (-95 to -100 ppm). The signal at -69 ppm results from unreacted clinker (alite). The overlap of the signals does not permit an unambiguous deconvolution.

The broad feature at -110 ppm is characteristic of silica fume not reacted during the hydration of the samples and quartz (Lothenbach et al., 2012).

The ²⁷Al-NMR spectra (also depicted in Figure 2) present two different signals at 63.0 and 16.7 ppm. Al in a tetrahedral environment- is generally observed between 50 and 80 ppm, while octahedral Al resonates between -20 and 20 ppm. Figure 2 shows that Al is in an octahedral and tetrahedral coordination. The observed tetrahedrally

coordinated ^{27}Al resonances are associated with the aluminum in the bridging position of the C-S-H phases and feldspar and the octahedral ^{27}Al to the presence of ettringite with probably some substitution of the Al by Fe.

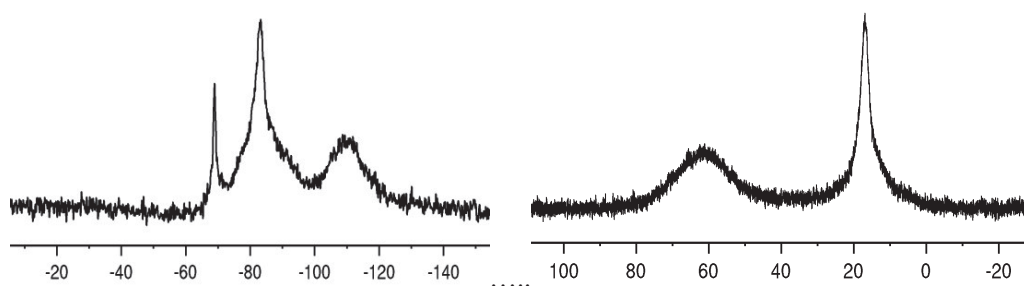


Figure 2: ^{29}Si MAS NMR spectra (left) and ^{27}Al MAS NMR spectra of the cement paste.

SURREY

The bulk composition of the reference mix design binder used to manufacture the RPM was analysed by X-ray fluorescence, the results of which are summarised in Table 7.

Table 7: XRF data providing the bulk composition of the binders used to form the Cebama reference paste and concrete.

Binder	CaO (%)	SiO ₂ (%)	Al ₂ O ₃ (%)	SO ₃ (%)	MgO (%)	Fe ₂ O ₃ (%)	K ₂ O (%)	TiO ₂ (%)	MnO (%)
Silica Fume	1.46	93.10	1.44	0.47	0.88	0.91	1.73	-	-
CEM I 42.5	67.72	17.60	3.42	3.81	0.6	5.17	1.3	0.17	0.21
Blast furnace slag	43.13	32.3	9.85	3.68	7.40	0.74	1.20	1.36	0.34

Blocks have been cast to determine the interaction of the Cebama reference paste with three selected groundwaters: granitic, saline (Gascoyne, 2002) and clay (Vinsot et al., 2008). Changes in groundwater composition will be measured over an extended period and cement samples will be taken periodically to observe mineralogical changes to existing phases and the formation of new phases. Analyses will be performed utilising XRD, SEM, RAMAN, EXAFS and XANES.

CTU - UJV

Laboratory work is based on ageing procedures among Czech bentonite, low-pH binder and groundwater from the underground laboratory Josef under high temperature. The ageing procedures on low-pH reference paste samples are ongoing since December 2016. Laboratory analyses of cementitious materials mainly include uniaxial strength test on thin samples, mineralogy, pH of leachates and diffusion properties. More detail can be found in CTU and UJV contribution to the 2nd CEBAMA Annual Workshop (Vašiček et al., 2017).

Because the interaction occurs mainly on interfaces (i.e., cement sample surface), specific shape of the samples is used to magnify this effect when uniaxial strength is tested. Thin plates (cylinders with diameter of 50.0 mm and height of 8.2 mm) and corresponding non-standard punch test method are being used. Due to specific shape and large amount of samples needed CTU decided to prepare them at their own laboratory according to procedure provided by VTT. Therefore, the first step focused on testing of eight manufacturing alternatives. The tests varied in plasticizer and various details in mixing procedure (two types of plasticizer from three sources were used - Ha-Be supplied by VTT, Ha-Be purchased on market and alternative one by DenBraven; procedure varied in total

amount of mixed material, duration of pouring into the forms and time intervals between adding of particular components).

All mixing tests provided thin samples for punch compressive strength test. At least nine samples were used as one sample set. Results (standard variation of datasets 3 - 12%) were recalculated to the cubic shape of the sample and plotted in Figure 3. Test no. 8 provided input values for future comparison of results from ageing procedures.

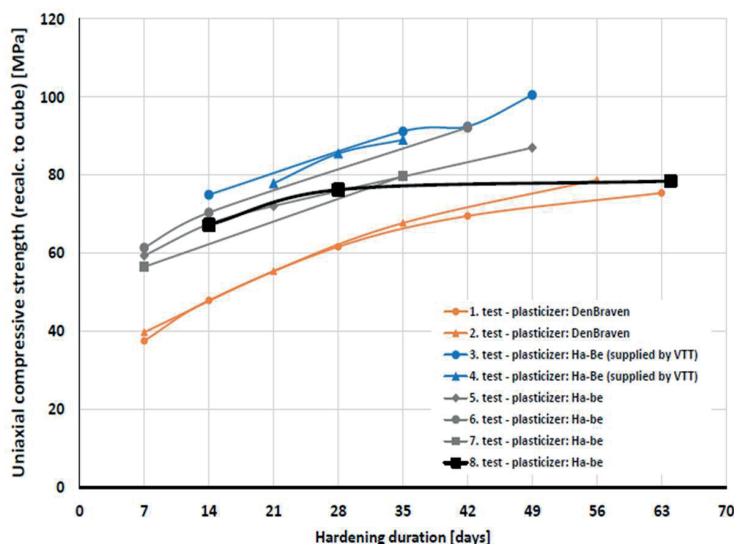


Figure 3: Uniaxial compressive strength on thin samples (recalculation to cubic shape). Comparison of 8 sets of mixing tests with variations in plasticizers and mixing procedure details.

Up to now, only the first measurement of cement paste pH in leachate is complete. Preliminary pH leachate tests (after 170 days since LPC mixing) were performed following the procedure described in (Alonso et al., 2012) and are shown in Table 8. First sampling of the reference paste samples is expected after 9 months of interaction - in September 2017. Then all of the analyses described in Table 2 will be performed.

Table 8: pH values of Cebama reference paste samples at different conditions (170 days after mixing).

paste sample	pH in suspension	pH in leachate
Curing 10°C	12.4	12.4
Josef GW 10°C	12.3	12.3
Josef GW 95°C	11.1	11.0

Note: Josef GW - groundwater from the Josef Underground Facility

VTT

VTT measured fresh-stage properties of the Cebama reference paste. Cement paste workability was characterized using a Haegermann flow table. Haegermann flow of the reference paste was 190 mm (DIN EN 1015, 2007). Compression strength, density and pore solution pH of the reference paste is presented in Table 9.

Table 9: Compression strength, density and pore solution pH of RPM at various ages. Pore solution pH was measured according to method described in the literature (Alonso et al., 2012).

Age	Compression strength	Density	pH
7 d	72.0 MPa	2,100 kg/m ³	-
28 d	111.0 MPa	2,150 kg/m ³	12.22
423 d	-	-	11.56

JUELICH

Complementary to experiments addressing the diffusion of safety relevant radionuclides in the Cebama reference paste, JUELICH characterized its microstructure and phases distribution. Here, we focused on the identification of phases and reductive species providing potential sorption sites for the radionuclides under investigation. Single components of the binder (quartz filler, silica fume, blast furnace slag and CEM I 42.5 MH/SR/LA) were analysed by SEM/EDX and XRD for their chemical and mineralogical composition. Scanning electron microscopic (SEM) investigations including SEM/EDX mapping and XRD were performed to analyse the microstructure of and phase distribution about 400 days after casting of the samples. XRD data on the grinded VTT reference paste show, beside intense reflexes of the quartz filler, only weak reflexes of crystalline hydration phases, mainly ettringite, and amorphous C-S-H. The microanalytical investigations reveal the presence of unhydrated clinker material, mainly C₂S, unreacted quartz filler, and blast furnace slag (Figure 4). The silica fume was found to be the main source of carbon present in the reference cement paste. Iron rich phases such as Fe-oxides and -sulphides were identified that can provide for the immobilisation of redox-sensitive radionuclides in the hardened cement paste. The generated data on the phase distribution in the reference paste will be further processed to provide pseudo-colour 2D maps of the phase distribution.

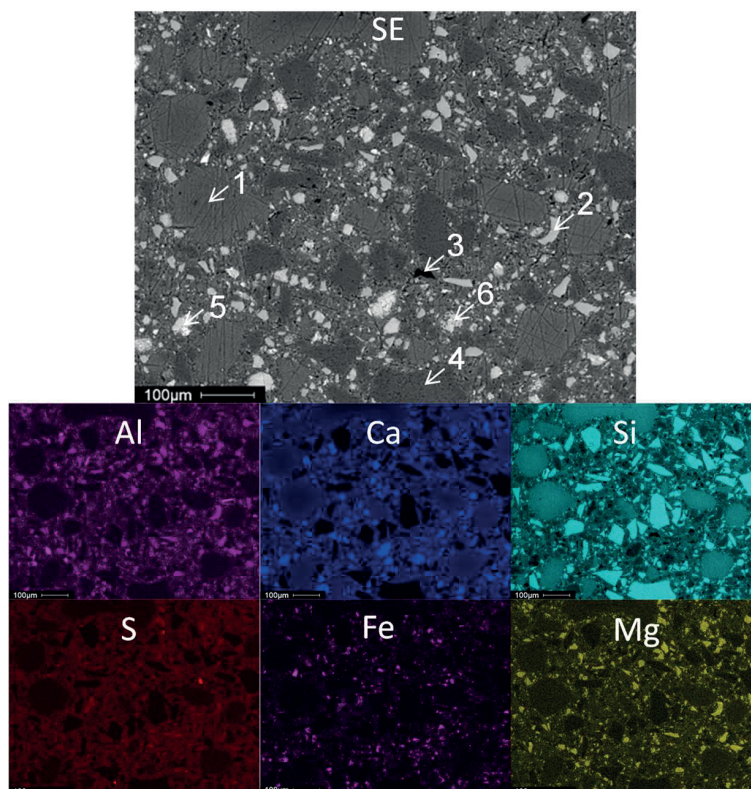


Figure 4: SEM/EDX mapping of the Cebama reference paste. 1) fumed silica, 2) blast furnace slag, 3) carbon, 4) quartz, 5) iron oxide, 6) unhydrated clinker.

Conclusions

Chemical characterization of the RCM determined by CSIC using XRD and EDX shown that portlandite and sulfoaluminate phases, such as ettringite, were not present. The EDX performed by CSIC reveal a low CaO/SiO₂ ratio = 0.55 ± 0.17 and enrichment in Al, CaO/Al₂O₃ ratio = 8 ± 4.6 .

According to the pH measurements in CSIC, the pore solution pH of the reference concrete (pH = 11.47) was below the saturation pH of calcium hydroxide (pH = 12.40). In VTT's measurement the reference concrete pH was 11.47 at the age of 428 days which is in excellent agreement with the CSIC results.

Regarding transport parameters, MIP confirms the low total porosity in RCM (2.3%) with pores distributed below 0.02 μm , (mean pore size 0.01 μm), all this agree with the high density of this concrete (2,420 kg/m³) measured by VTT and CSIC. The compression strength measured by VTT is 88.5 MPa after 424 days of aging. The concrete also present a high resistance to percolation of water, hydraulic conductivity 10^{-13} to 10^{-14} m/s and the pH of eluted water was close to neutral and far from the alkaline plume of OPC (Cau Dit Coumes et al., 2006).

Chemical characterization of the RPM determined by JUELICH and KIT identified ettringite in the paste samples. The observations further support the anticipated lower reaction degree of the paste samples compared to the concrete samples.

Reference paste pH measured in CTU-UJV presented pore solution pH 12.4. VTT measured pore solution pH 11.56 which is in agreement with the pH measured in KIT (pH = 11.7). CTU-UJV performed experiments in 10°C temperature whereas VTT and KIT experiments were performed in room temperature.

Future work

Concrete (RCM)

CSIC will study the evolution of hydration at further ages, evolution of interaction with granite and clayey groundwaters through percolation tests and transport Cl diffusion through natural tests, using RCM and RCM-LD-samples.

VTT will perform leaching experiments to the Cebama reference concrete, Binary- and Ternary- low-pH concretes.

KIT will characterize the chemical and microstructure of the concrete sample.

Paste (RPM)

KIT will study the diffusion of ³⁶Cl, ¹²⁹I, HTO and Be on RPM.

SURREY will use radioactive tracers (³H, ¹⁴C, ³⁵S and ³⁶Cl) to provide information on reaction mechanisms and kinetics governing incorporation of target elements into the cement matrix. Autoradiography will be used to measure migration rates of the radioactive tracers. Advection experiments will be carried out as described in (Felipe-Sotelo et al., 2016) where the radioactive tracers can allow an unambiguous determination of the uptake and movement of ions of interest.

Additionally, sorption / desorption experiments are in progress to study the uptake / retention of selected radionuclides (³⁶Cl, ⁷⁹Se, ¹²⁹I). These experiments should allow the determination of uptake and release kinetics

as well as the partitioning of the radionuclide between solid and solution. Analysis of the solids by XRD, SEM and EXAFS will provide insight to the distribution of radionuclides on and within the phases present.

These experiments will provide an understanding of the mobility of the target radionuclides through the paste. Through-diffusion experiments are being carried out in the manner described by Felipe-Sotelo et al. (2017). These experiments involve cylindrical blocks being cast, a well drilled into the centre of each block and the radionuclide of interest spiked into the central well. The well is then sealed and the block placed in equilibrated water. Breakthrough of the radionuclide is measured by monitoring the surrounding equilibrated water and distribution of radionuclide within the cement can be determined by autoradiography once the experiment has concluded.

JUELICH will study small monolithic samples of the Cebama reference paste in in-diffusion experiments using ^{226}Ra , $^{99}\text{Tc(VII)}$, molybdate and iodide; the effective diffusion coefficient of the paste will be determined by diffusion experiments with HTO in cooperation with SURREY.

UAM will perform mineralogy analysis using XRD with Rietveld approach and thermal analysis.

They will also conduct a small transport experiment including FEBEX-bentonite.

Acknowledgement

The research leading to these results has received funding from the European Union's Horizon 2020 Research and Training Programme of the European Atomic Energy Community (EURATOM) (H2020-NFRP-2014/2015) under grant agreement n° 662147 (CEBAMA).

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