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Abstract Portland cement paste is proposed as the material to backfill the annulus between the casing and the geological formation of a deep borehole repository for spent sealed radiation sources in Brazil. The cement paste is intended to function as structural material, an additional barrier against the migration of radionuclides outside the repository, and as a blockage against the transport of water between the different strata of the geological setting. The objective of this research is to investigate the behavior of the cement paste and to estimate its service life. In this paper we present the results of mechanical strength measurements and chemical and mineralogical analysis of samples to detect the changes caused by radiation, temperature and aggressive chemicals of groundwater to which the material will be exposed. Methods of analysis included Inductively Coupled Plasma Atomic Emission Spectroscopy, Ion Chromatography, X-Ray Diffraction, and Thermo-Gravimetric Analysis.

1. Introduction

The Nuclear and Energy Research Institute (IPEN-CNEN/SP), in São Paulo-Brazil, stores disused sealed radioactive sources (SRS) as radioactive waste. Many sources have long-lived radionuclides and high activity. The inventory amounts to tens of thousands sources and the total activity reaches hundreds of terabecquerels (Vicente *et al.*, 2004). Final disposal of this kind of waste is an unresolved issue in Brazil and a difficult problem in most countries. Shallow boreholes and shallowground disposal sites for low- and intermediate-level wastes cannot accept disused sealed sources for disposal and intermediate depth boreholes may be unacceptable for large inventories and in humid climate sites (Nel, 2004).

In order to find an alternative for disposal these sources, the Radioactive Waste Management Laboratory (RWL) at IPEN-CNEN/SP is developing a concept of disposition in deep boreholes, where SRS could be isolated from the human environment by the millennia that are needed by those sources to reach an acceptably low radiation risk (Vicente *et al.*, 2004).

In this concept, a borehole is drilled to a depth of a few hundred meters in a granite batholite, encased with a steel pipe and cemented by pumping down Portland cement-water slurry, which is left to harden in place, backfilling the annular space between the steel casing and the geological formation. The hardened cement paste is intended to function as an additional barrier against the migration of radionuclides toward the biosphere and as a blockage to hinder the flow of water between different layers of the geological setting crossed by the borehole (Milodowski *et al.*, 2013).

The long-term safety of this concept relies on multiple engineered and natural barriers. The overall performance of the whole system depends on the behavior of all barriers, their interactions with the disposed wastes and the components of the environment, and their evolution over time. Of all components of the system, the cement paste is the material with the least known behavior in the long term (L'Hostis *et al.*, 2008; Mehta *et al.*, 2008; Scrivener & Kirkpatrick, 2008; Xie *et al.*, 2008). The complex chemistry of Portland cement and the variability of wastes and repository conditions is a possible explanation for the persisting question of the long term behavior of cementitious materials under repository conditions.

In the repository for sealed sources, the cement paste will be exposed to some factors that are deemed to affect negatively its durability. Attempts to investigate the degradation of cement paste under several conditions have been undertaken by various authors, exposing the cement paste or mortar to some factors, as higher temperatures and pressures (Cheng *et al.*, 2012; Damidot *et al.*, 2011; Le Saoût *et al.*, 2006; Lothenbach *et al.*, 2008), aggressive chemicals dissolved in the groundwater (Adamopoulou *et al.*, 2011; Bullard *et al.*, 2011; Deby *et al.*, 2009; Kaminskas & Barauskas, 2012; Loser *et al.*, 2010; Lothenbach *et al.*, 2010; Ukrainczyk *et al.*, 2012), and the radiation field of the radioactive wastes (Bouniol & Bjergbakke, 2008; Bouniol, 2010; García Calvo *et al.*, 2010; NAGRA, 2008; Vodák *et al.*, 2005). However, there is a lack in the study of synergetic effects in long term.

The present research aimed at investigating the durability of cement paste under repository conditions using accelerated tests. Specimens of cement paste were exposed to the high levels of the stressing factors foreseeable at the depth of the repository in order to evaluate the exposure effects on the cement paste by observing changes in their chemical, structural, and mineralogical characteristics.

2. Materials and Methods

The behavior of the cement paste exposed to aggressive conditions was investigated using accelerated tests in laboratory. Cement paste samples are examined after being exposed to the environmental conditions that are expected to prevail in the repository environment and the results are compared with those obtained with unexposed specimens or specimens exposed to reference conditions. The following exposure conditions were selected:

a) Immersion in salt solution (SS), distilled water (DW), or kept in dry storage (DS);
b) Room temperature (20°C) or high temperature (60°C);
c) Immersion time of 30 days (30D) or 60 days (60D) (not for dry storage);
d) Irradiation to a dose of (400 kGy) or background radiation (0 kGy).

Twenty four sets, with five cubic cement paste specimens (cps) (20 x 20 x 20 mm) each one, were cast and left to set inside resin molds, for one day. They were demolded, cured in saturated limewater for more six days and then stored under the above conditions used to accelerate stress effects.

The cps were cast with Portland Cement Type V, according to Brazilian specifications, which is equivalent to the HES cement of ASTM specifications (ASTM, 2007) with a water to cement (w/c) ratio of 0.35.

Tests were planned as a complete multi-factorial experiment. Table 1 shows the assignment of each sample set to the exposure conditions. Sets identified by U and V functioned as reference base line to which the other sample results were compared.

After exposure to the stressing conditions, the effects of each factor on the cps were observed by changes in their characteristics. Compressive strength tests were performed on all cps and some of them were investigated in terms of changes in mineralogy by X-ray diffraction (XRD) and thermo-gravimetric analysis (TGA).

Immersion solutions were prepared with the cations and the anions usually present in a typical granitic-environment groundwater. The composition of salt solutions are showed in Table 2. After the assays, the resulting solutions were analyzed by ICP-OES (Ca, Na, K, Mg, and Si) and ion chromatography (SO_4^{2-}) to detect leaching of chemical species from, or penetration into cement paste.

To kept the cps at dry storage, they were carefully placed in plastic pots and kept at environmental humidity, around 50%.

A multipurpose compact irradiator with 3,4 TBq of ^{60}Co, was employed to irradiate the samples to accumulate a radiation dosis of 400 kGy what lasted about 100 hours.

Table 1. Sample set ID and test conditions assignment

Sample Set ID	**Immersion/ dry storage**			**Temperature**		**Immersion time**		**Irradiation**	
	DW	SS	DS	20°C	60°C	30D	60D	0 kGy	400 kGy
A	*			*		*		*	
B	*			*			*	*	
C		*		*		*		*	
D		*		*			*	*	
E	*				*	*		*	
F	*				*		*	*	
G		*			*	*		*	
H		*			*		*	*	
I	*			*		*			*
J	*			*			*		*
K		*		*		*			*
L		*		*			*		*
M	*				*	*			*
N	*				*		*		*
O		*			*	*			*
P		*			*		*		*
Q			*	*			*	*	
R			*	*			*	*	
S			*		*	*		*	
T			*		*		*	*	
U			*	*			*		*
V			*	*			*		*
W			*		*	*			*
X			*		*		*		*

Table 2. Sample set ID and test conditions assignment

Ionic Species concentration (in mg/L)									
Ca	Na	K	Mg	Cl	HCO_3	SO_4	Si	Fe	NO_3
$1{,}79.10^{0}$	$1{,}3.10^{0}$	$3{,}3.10^{-1}$	$5{,}59.10^{-2}$	$4{,}9.10^{0}$	$6{,}40.10^{-2}$	$5{,}50.10^{-1}$	$1{,}00.10^{-4}$	$1{,}31.10^{-4}$	$1{,}72.10^{0}$

3. Results

The medians, maximum and minimum of the mechanical strength of all cps sets are presented in Figure 1. Student's t-test, at the 0.05 level of significance, was used to assess the significance of differences between the mechanical strength of cps submitted to each treatment in comparison to reference cps. The results of all cps submitted to the same treatment level of each factor were analyzed as one group, in order to evaluate the effects of each treatment individually. The results of the t-test are presented in Table 3.

It was observed that the immersion in both SS and DW increased the mechanical strength of samples, as did the temperature rise, most probably because the hydration degree of samples was incomplete in early ages and was completed during immersion. SS, however, lowered the mechanical resistance, by interactions of the cement paste with the chemical species present in the SS.

Table 3. Mechanical strength and results of t test for each treatment

Treatment		Mechanical Strength (MPa)	Statistical significance
Immersion	SS or DW DS	117 ± 17 89 ± 11	Extremely significant
Immersion solution	DW SS	123 ± 13 111 ± 17	Very significant
Temperature	20°C 60°C	104 ± 18 112 ± 22	significant
Immersion time (days)	30 days 60 days	106 ± 21 110 ± 20	not significant
Irradiation dose (kGy)	0 400	107 ± 20 109 ± 21	not significant

Statistical significance: : p-value < 0.0001 – extremely significant; 0.0001 > p -value > 0.0100 – very significant; 0.0100 > p -value > 0.0500 significant; 0.0500 > p -value - not significant

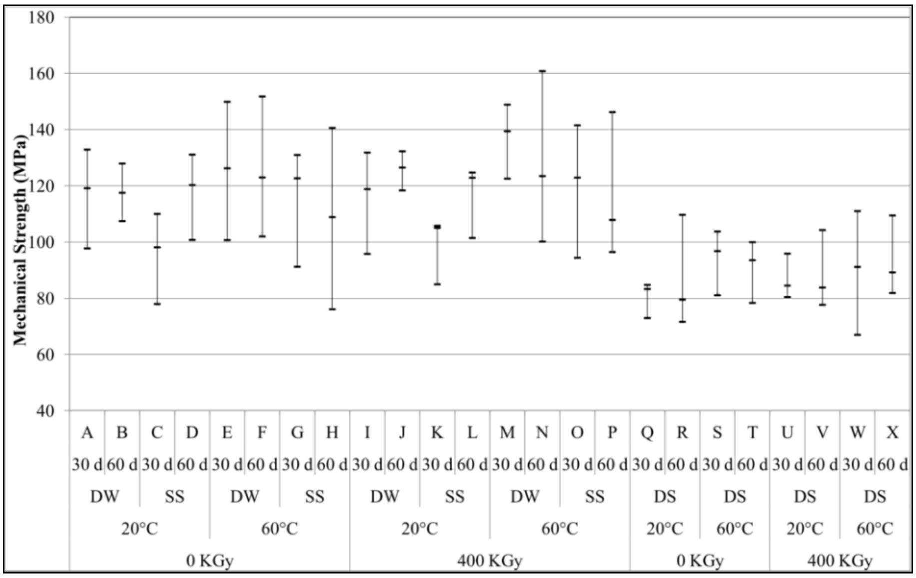

Figure 1. Mechanical strength maximum, minimum and median of each set of cubic cps

The cps that were kept at DS and at the higher temperature showed weight loss, caused by evaporation of the free water present in the cement pores. Cps immersed in DW or in SS had both a gaining of mass by absorbing water from the solution, which however was lost to the environment, after a while as expected (Lécolier *et al.*, 2007; Galíndez & Molinero, 2010). However, the cps immersed in SS presented a residual gain of mass, caused by precipitation or by chemical reaction of solution salts with cement compounds.

Changes in the concentrations of chemical species in the immersion solutions are shown in Figure 2. The concentrations of Al, Fe, NO_3^-, Cl^- and F^- varied only slightly while the other presented a marked difference.

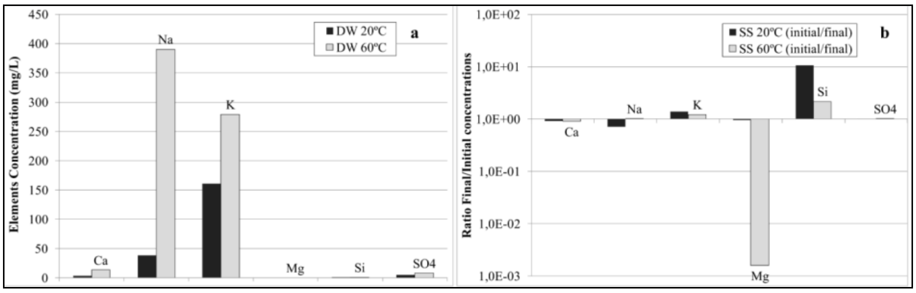

Figure 2. a) Concentration of species after immersion of cubic cps in distilled water; and b) initial/final ratio in concentration of species after immersion of cubic cps in salt solution

As expected, Ca leached from cement paste under immersion in DW and was incorporated in cps submitted to SS immersion; calcium is absorbed by C-S-H or react with bicarbonate to produce calcite. Si concentrations changes were not relevant, once the initial concentration in SS was small. Alkalis (Na and K) were released from C-S-H sites and leached to solution, inducing the penetration of Ca and Mg. Sulfate penetration was low, indicating that the delayed ettringite formation in the cement paste did not occur.

Samples from sets of cps, randomly selected and representative of all treatments, were separated, grinded to a fine powder in agate mortar, and analyzed by XRD.

A diffractogram of the samples is presented in Figure 3. In general, the XRD patterns indicate no difference in the mineralogy, except by the ettringite peaks (1, 2 and 4) that are missing in specimens kept in DS at 60°C (sets S, T, W and X). A semi quantitative analysis, by the Reference Intensity Ratio (RIR) method, confirmed this statement. Furthermore, cps kept in immersion showed higher degree of hydration (lower belite and alite quantities) than those that were kept in dry storage.

The spectra were compared using cluster analysis. A dendrogram program calculates a similarity matrix, transforms similarity coefficients into distances and makes a clustering using the Unweighted Pair Group Method with Arithmetic mean (UPGMA) algorithm. The method was able to group sample sets that were exposed to similar treatments, showing that samples submitted to the same treatments have similar mineralogical composition.

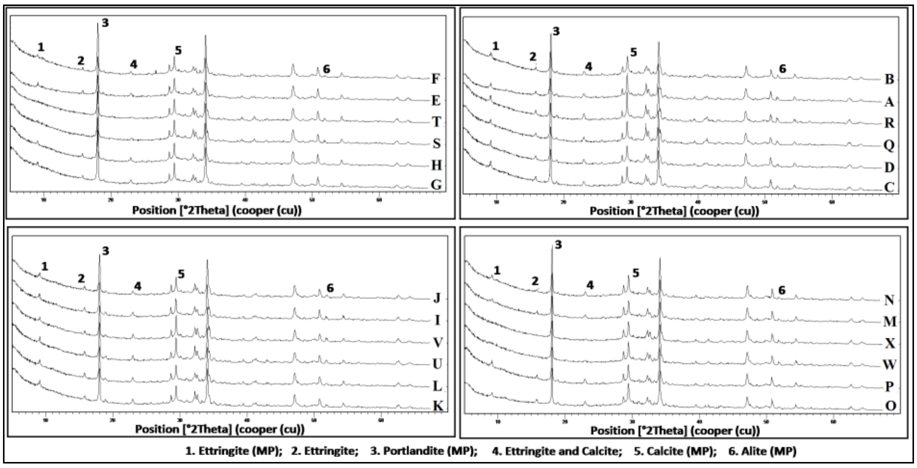

Figure 3. XRD diffractograms of the selected cement paste samples

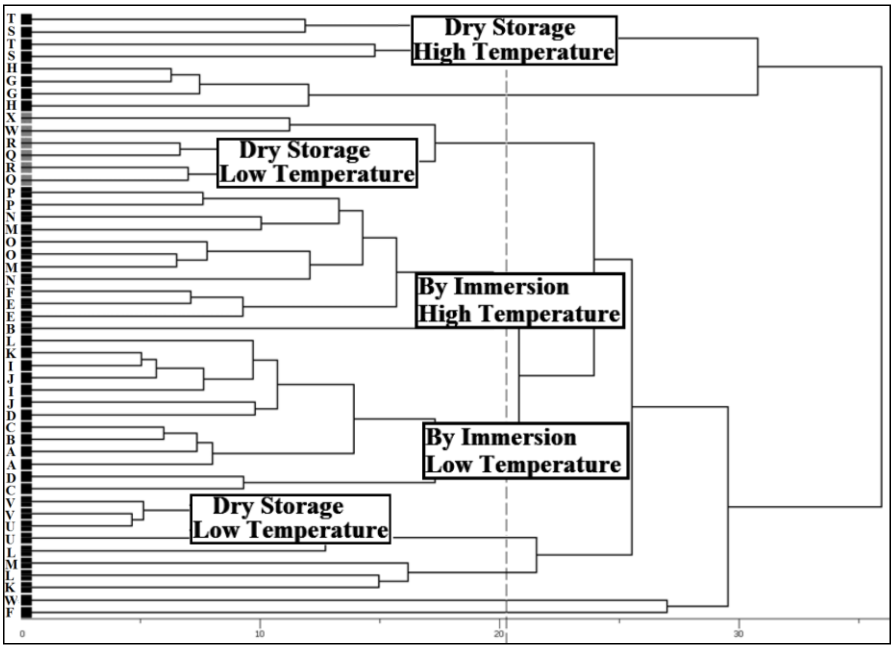

Figure 4. Dendrogram obtained by cluster analysis of diffractograms of cement samples

Six cement paste samples were analyzed by TGA after irradiation and immersion. Table 3 shows the percent loss of weight of each sample at the indicated temperatures. The weight loss at each temperature zone shows the decomposition of some mineral compounds or loss of pore water. In Table 4 it is possible to see that samples kept in DS at 60°C showed the lowest weight loss between 25°C and

290°C, indicating low quantities of ettringite and/or pore water (Alarcon-Ruiz *et al.*, 2005).

Table 4. Weight loss percentage of each sample at the indicated temperatures.

TGA Sample ID	Sample Set ID	Storage Conditions	Irradiated	Weight Loss (%)				
				25 to 60°C	60 to 290°C	290 to 485°C	485 to 1000°C	Residues at 1000°C
1	Q	DS; 20°C; 60 days	No	4,5	10,8	5,0	4,9	74,5
2	U	DS; 20°C; 60 days	Yes	4,4	11,0	4,8	5,1	74,6
3	S	DS; 60°C; 30 days	No	3,2	8,6	5,1	4,1	79,0
4	W	DS; 60°C; 30 days	Yes	3,1	9,0	5,5	7,5	74,9
5	C	SS; 20°C; 30 days	No	4,4	11,9	4,9	6,2	72,7
6	A	DW; 20°C; 30 days	No	3,7	11,5	5,0	6,4	73,3

4. Discussion

Many authors in the literature shows that the mechanical strength of cps submitted to immersion is lower than that of cps kept in DS. However, it was observed the opposite effect for both SS and DW immersed CPS. This behavior was the result of incomplete hydration of the CPS at the start of the immersion tests (Lee *et al.*, 2005). Ettringite formation in early ages fills the cement paste pores increasing the resistance. On the other hand, in later ages, ettringite formation causes cracks and spalling of cement paste (Al-Amoudi, 1998; Collepardi, 2003; El-Hachem *et al.*, 2012; Torii *et al.*, 1995). Cps immersed in SS presented lower resistance than cps immersed in DW, probably caused by magnesium penetration into cement paste. According to Bénard *et al.*, (2008), the presence of some chemical species in solution could affect the hydration process by poisoning, adsorption or precipitation.

During the cure, the hydration was not complete and this process was restarted when cps were immersed, leading to an increase in the mechanical strength. It was also observed by Aziz *et al* (2005), who investigated the relationship between hydration process and durability of cement blends, proving that the mechanical strength is higher in samples kept for a longer hydration time. In fact, XRD semi-quantitative analysis revealed that cps kept in DS presented more quantities of anhydrous cement compounds than immersed ones.

The attack of aggressive chemical species in immersion solutions was observed as the leaching of Na^+ and K^+ from cement paste to solution and the penetration of Ca^{2+} and Mg^{2+} into cement paste. Decalcification and delayed ettringite formation were not detected once Ca^{2+} was not leached, neither SO_4^{2-} penetrated into cement paste. The reaction of bicarbonate with calcium, that can produce calcite, blocking the pores and reducing permeability, was not observed at all (Berner, 1992; Matschei & Glasser, 2010). Marumo (1997) investigated the sulphate penetration into cement specimens immersed in simulated groundwater and observed a small relative penetration of this ion in contrast to specimens immersed in solution with higher concentrations (Marumo, 1997).

The XRD cluster analysis could identify changes in the mineralogy caused to exposed samples. However, except by ettringite differences were tenuous and hardly observed. The absence of ettringite in cps that were kept in DS at 60°C could be explained by decomposition. Although the temperature of decomposition is about 110-120°C, it may be initially decomposed to meta-ettringite, an amorphous compound, above 50°C in the absence of water (Meller *et al.*, 2009; Zhou *et al.*, 2004; Zhou & Glasser, 2001). The ettringite present in the cps kept in dry storage at 20°C confirms that it was initially formed and then decomposed at 60°C.

Cps kept in dry storage showed significant weight loss after exposure, mainly those kept at 60°C. This behavior is expected once the loss of water is a natural process in DS while cps immersed absorbed water and gained mass. In fact, lower quantities of pore water in cps kept in dry storage were confirmed by TGA.

Although high temperature damages the cement, this rise is beneficial to cement resistance if occurs during curing time (Ballester *et al.*, 2009; Soroka, 1993). Once the hydration process results from chemical interactions, higher temperature may accelerate the reaction kinetics, resulting in higher mechanical strengths. Since the immersed cps were considered as under a continued hydration process, the higher temperature caused a slight but observable increase in the mechanical strength from 113 MPa (20°C) to 121 MPa (60°C) in average.

Influence of irradiation on the mineralogical composition was not observed when analyzed alone or in combination with possible synergic effect with other treatments. The radiation dose of 400 kGy was not capable to induce alterations in cement paste.

Time of exposure was not able to alter neither the mechanical strength, nor the mineralogy of cps. The time of exposurein future work will be significantly increased.

5. Conclusions

Durability of cement paste was investigated using accelerated tests in laboratory as a tentative approach to establish its service life under repository conditions. Short-term results obtained under laboratory conditions are analyzed and correlated with the intrinsic properties of the material. With the results obtained so far it was possible to conclude that:

a) After a period of immersion in water, Cps further hydrated and presented higher mechanical resistance, as expected. Chemically aggressive species present in SS degraded cement paste and mechanical strength reduced. However, the concentration of some ions in groundwater is too low to induce serious damage to cement paste;

b) Dry storage did not allow a complete hydration as a consequence of pore water evaporation. High temperatures intensified this process and led to the ettringite decomposition to meta-ettringite;

c) The higher temperature accelerated hydration kinetics and promoted higher mechanical resistance in cps kept under immersion;

d) The irradiation doses applied was unable to change the mineralogy of cps;

e) No statistically significant differences were observed between 30 or 60 days exposure time, for the test conditions.

Despite the parameters evaluated in this work did induce changes in mineralogy of cement paste, they were not able to damage specimens or lower drastically their mechanical resistance. A complete hydration of specimens during the curing process is an important factor to evaluate immersion and dry storage effects. Furthermore, longer period of time of immersion/storage and higher irradiation doses must be applied to induce mineralogy changes in cement paste. Further work is expected to allow extrapolating the short-term results obtained under laboratory conditions to the actual conditions in the repository over the long-term, as an attempt to determine de service life of the cement paste.

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