ASSESSMENT OF RADIATION AND TEMPERATURE LOADS ON CEMENT COMPOUND CONTAINING SIMULATED RADIOACTIVE WASTE

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The paper focuses on radiation effects of ionizing sources and elevated temperatures produced on cement compounds containing simulated high-level and intermediate-level LRW. The paper presents the identified physical and chemical properties of the cement compounds and their alterations under the influence of elevated temperatures and ionizing radiation with the absorbed dose of up to 10^8 Gy.

Keywords: *radioactive waste, simulated radioactive waste, radiation stability, dose rate, temperature loads, strength, radionuclide leaching rate.*

Radioactive waste (RW) poses a serious threat to the environment and humans and should be converted into a safe form suitable for disposal. Selection of appropriate final waste forms is not an easy task to accomplish, since many factors and combinations of various conditions have to be taken into account. For example, the conditioning process should be simple, reliable, safe, cost-effective and the final waste form should comply with waste acceptance criteria for disposal.

Vitrification and cementing technologies are most commonly applied in different countries around the globe to provide RW immobilization and to obtain suitable final waste form. As a rule, high-level waste is vitrified, whereas intermediate- and low-level waste is cemented [1].

Nevertheless, the widespread use of cement materials in various structures of nuclear facilities [2, 3] being exposed to high levels of ionizing radiation suggests the ability of cement compound to withstand high dose loads and its potential application as a matrix for high-level waste (HLW) immobilization.

This paper presents a follow up research [4—6] on the comprehensive assessment of changes in the standardized properties, phase composition and microstructure of cement compounds occurring under the influence of high doses of ionizing radiation equivalent to those emitted by HLW during its storage.

Current stage of research addresses heat release impacting cement compound hardening and various types of ionizing radiation affecting it involving simulated nitrate-containing high-level LRW (HLW simulator) and genuine nitrate-containing intermediate-level LRW. To address this task, mechanical, physical and chemical parameters of cement compounds hardening at elevated temperatures and cement compounds after being exposed to gamma-, alpha- radiation and electrons were studied.

Subjects and research methods

The studies involved samples of cement compounds fabricated from Portland cement (PC) either with some amounts of bentonite clay powder (DB) being added or with no additives and with a grouting fluid. Tap water, simulated HLW and genuine LRW at a water-cement ratio (W/C) of 0.5 were used as grouting fluid. Bentonite clay powder was introduced with the Portland cement and was taken into account under the W/C ratio as C = 95 % PC + 5 % DB.

Simulated HLW had a salinity of 530 g/l and a composition corresponding to the average composition of the waste generated at PA Mayak [7], g/l: NaOH — 196.8; $NaNO_3$ — 209.1; $NaNO_2$ — 108.1; $NaHCO_3$ — 17.64; $K_2Cr_2O_7$ — 10.82; KCl — 1.47; $Zn(NO_3)_2$ — 0.02; $Pb(NO_3)_2$ — 0.07; CsCl — 0.05; $SrCl_2$ — 0.5. Genuine LRW had the following composition, g/l: Cl^- — 9.2; NO_3^- — 71.1; ClO_4^- — 4.3; Na^+ — 187.2. LRW activity was associated with the following radionuclides, Bq/L: ^{137}Cs — $2{\cdot}10^6$, ^{90}Sr — $3{\cdot}10^6$, ^{239}Pu — $1{\cdot}10^5$, ^{152}Eu — $3{\cdot}10^5$. According to regulatory requirements, the LRW refers to the intermediate-level waste category.

Thus, the studies were performed based on cement compound samples indicated in Table 1.

Table 1. Cement compound compositions

Composition	Bonding agent	Additive	Grouting fluid	W/C
№ 1	PC	–	water	0.5
№ 2	PC	DB	water	0.5
№ 3	PC	–	Simulator, s/c 530 g/l	0.5
№ 4	PC	DB	Simulator, s/c 530 g/l	0.5
№ 5	PC	–	LRW	0.5
№ 6	PC	DB	LRW	0.5

PC — Portland cement, DB — bentonite clay powder,
W/C — water-cement ratio, s/c — salt content

To evaluate mechanical, physical and chemical properties of cement compounds, compressive strength was measured using standard methods and equipment described previously in [4, 5]. Solidification and hardening of cement compounds occurred under air-wet conditions both at a room temperature and at elevated temperatures of 50 and 90 °C, under prolonged exposure to water, alternating freezing/thawing cycles and radiation exposure.

The cement samples were exposed to radiation in a gamma chamber fitted with a ^{137}Cs radiation source of 661 keV. Exposure times corresponded to those required for the absorbed dose to reach 10^6, 10^7 and 10^8 Gy, respectively. Electron exposure was studied using UELV-10-10-S-70 linear electron accelerator characterized with the following parameters: energy — 8 MeV, pulse duration — 6 µs, pulse repetition frequency — 300 Hz, average beam current — ⩽ 800 µA, sweep width — 245 mm, sweep frequency — 1 Hz. Depending on how fast the sample passed through the electron beam, the dose rate averaged 1.2 kGy/s. Irradiation with alpha-emitters was carried out by injecting the ^{239}Pu radionuclide (a nitric acid solution) directly into the grouting fluid. The dose rates of 10^6, 10^7, 10^8 Gy in the cement compound samples were reached following their exposure for 90 days under activity of ^{239}Pu per compound sample of $3.5{\cdot}10^8$, $3.9{\cdot}10^9$ and $2.4{\cdot}10^{10}$ Bq, respectively.

Radionuclides leaching rate was evaluated according to a standard method [8] in a thermostat under static conditions at a temperature of 25 °C. Deionized water (with a conductivity of 18 Mohm/cm) was used as a leaching medium. To evaluate the content of Cs, Sr, Pu radionuclides in leach waters, a gamma spectrometer with a high-purity germanium detector Canberra GC 3020 (Canberra, USA) and a Tri-Carb 2810T liquid scintillation spectrometer (Perkin Elmer, USA) were used.

Mechanical, physical and chemical property indicators of the compounds were identified according to the following flow chart: compressive strength — hardening time of 7, 14, 28 days; frost resistance — hardening time of 28 days; water resistance of samples (resistance to prolonged exposure in water for 90 days) — from the hardening time of 28 days; radiation resistance of the samples and radionuclide leaching rate — from the hardening time of 28 days. Measurements and tests involved cube — (2 × 2 × 2 cm) and beam — (1 × 1 × 3 cm) shaped samples.

Results and discussion

Tables 2 and 3 present the data on physical and mechanical properties and estimated leaching rates for unirradiated cement compounds. They also present the compressive strengths of samples that have gained strength at elevated temperatures, after being subject to 30 cycles of freezing/thawing and exposed in an aqueous medium. All the values identified are average values obtained from two parallel measurements.

Measured compressive strengths of cement compound samples, the hardening of which took place at temperatures of 50 and 90 °C, did not show any negative effects of elevated temperature both on the common solidification process and the values being considered typical for the strength development periods. The strength of the compounds

***Table 2.** Mechanical compressive strength of cement compounds that gained strength at different temperatures following frost and water resistance tests*

Composition	Curing temperature, °C	Mechanical strength of the compound, MPa (average measurement)						
		Hardened under air-wet conditions			During frost resistance testing		During water resistance testing	
		Day 7	Day 14	Day 28	Control compound with similar hardening age	Following 30 freezing/thawing cycles	Control compound with similar hardening age	After being held in water for 90 days
1	25	17.9±4.5	26.4±6.6	33.8±8.5	35.7±8.9	33.8±8.5	41.4±10.4	35.8±9.0
2		16.8±4.2	25.0±6.3	32.7±8.2	35.2±8.8	33.1±8.3	41.1±10.3	35.1±8.8
3		13.3±3.3	21.2±5.3	27.7±6.9	29.1±7.3	26.8±6.7	32.5±8.1	27.6±6.9
4		12.2±3.1	20.0±5.0	27.2±6.8	28.4±7.1	26.5±6.6	32.0±8.0	27.1±6.8
1	50	18.3±4.6	26.9±6.7	34.7±8.7	35.9±9.0	34.0±8.5	41.6±10.4	35.7±8.9
2		17.1±4.3	26.2±6.6	33.8±8.5	35.6±8.9	33.8±8.5	41.5±10.4	35.5±8.9
3		14.9±3.7	22.8±5.7	29.1±7.3	30.0±7.5	27.3±6.8	33.0±8.3	27.2±6.8
4		13.5±3.4	21.3±5.3	28.2±7.1	29.7±7.4	26.9±6.7	32.9±8.2	27.1±6.8
1	90	19.2±4.8	29.9±7.5	35.5±8.9	36.2±9.1	34.4±8.6	40.9±10.2	35.3±8.8
2		19.6±4.9	28.3±7.1	34.8±8.7	35.8±9.0	34.1±8.5	40.7±10.2	34.9±8.7
3		15.5±3.9	23.5±5.9	30.6±7.7	31.3±7.8	28.5±7.1	33.2±8.3	28.1±7.0
4		14.4±3.6	23.1±5.8	29.5±7.4	31.0±7.8	28.0±7.0	33.1±8.3	28.4±7.1

***Table 3.** ^{137}Cs, ^{90}Sr and ^{239}Pu leaching rates at the 28th day of the experiment involving samples have gained their strength under different temperatures*

Composition	Curing temperature, °C	Leaching rate, g/cm²·day		
		^{137}Cs	^{90}Sr	^{239}Pu
1	25	$7.1 \cdot 10^{-5}$	$9.7 \cdot 10^{-6}$	$3.1 \cdot 10^{-6}$
2		$5.6 \cdot 10^{-5}$	$8.4 \cdot 10^{-6}$	$9.6 \cdot 10^{-7}$
3		$2.2 \cdot 10^{-4}$	$4.5 \cdot 10^{-5}$	$5.8 \cdot 10^{-6}$
4		$8.3 \cdot 10^{-5}$	$2.7 \cdot 10^{-5}$	$5.1 \cdot 10^{-6}$
1	50	$2.5 \cdot 10^{-5}$	$5.1 \cdot 10^{-6}$	$1.2 \cdot 10^{-6}$
2		$1.8 \cdot 10^{-5}$	$3.7 \cdot 10^{-6}$	$8.9 \cdot 10^{-7}$
3		$9.1 \cdot 10^{-5}$	$1.3 \cdot 10^{-5}$	$4.0 \cdot 10^{-6}$
4		$6.3 \cdot 10^{-5}$	$9.3 \cdot 10^{-6}$	$4.8 \cdot 10^{-6}$
1	90	$3.4 \cdot 10^{-5}$	$3.1 \cdot 10^{-6}$	$9.1 \cdot 10^{-7}$
2		$2.1 \cdot 10^{-5}$	$1.8 \cdot 10^{-6}$	$7.5 \cdot 10^{-7}$
3		$7.0 \cdot 10^{-5}$	$1.0 \cdot 10^{-5}$	$2.2 \cdot 10^{-6}$
4		$3.9 \cdot 10^{-5}$	$7.2 \cdot 10^{-6}$	$2.6 \cdot 10^{-6}$

varied over time depending on their composition and growed due to hydration processes occurring in the cement stone. These results generally correlate with previously published studies referring to the influence of ambient temperature on the cement stone hardening [9, 10].

The following points can be emphasized based on the results obtained. Strength development of cement stone accelerates with an increase in the curing temperatures, provided that the required humidity is maintained in the thermostat. It was demonstrated that under these conditions, bentonite clay powder addition cannot produce any pronounced effect on the strength characteristics. The strength of cement compound samples involving simulated HLW appears to be 20—25% lower as compared to cement compounds produced by mixing with distilled water.

Under considered conditions, cement compounds meet the requirements [11] on the acceptable strength of cemented waste (5 MPa) starting from a hardening time of 7 days. Moreover, strength loss due to freezing/thawing and prolonged exposure to water does not exceed 25% compared to the control sample having the same hardening age. In terms of strength, the tested samples meet the acceptance criteria [12] for RW class 2 — not less than 10 MPa with a test-driven strength loss of no more than 20% of the established value. Thus, the considered cement compounds comply with strength criterion specified by relevant regulations governing activities at nuclear facilities.

Basically, cement compounds involving bentonite clay additive (No. 2 and No. 4) differ from those with no additives (No. 1 and No. 3) by lower ^{137}Cs, ^{90}Sr and ^{239}Pu leaching rates (with an up to 30% discrepancy) (see Table 3), which is also true for samples that gained their strength at temperatures of 50 and 90 °C. It may be also noted that during cement compound hardening, an increase in the ambient temperature of up to 90 °C produced no negative effect on the chemical resistance: no increase in the radionuclide leaching rate was observed.

To evaluate the radiation resistance, cement compounds were exposed to various types of ionizing radiation. Subsequently, cement compound samples were tested to measure their frost and water resistances, as well as the radionuclide leaching rate.

Table 4 presents the experimental data on the strength of the samples after their exposure to gamma radiation and electrons.

Table 4. Mechanical compressive strengths of cement compounds subjected to radiation exposure

Composition	Mechanical strength of the compound, MPa					
	Gamma-quants			Electrons		
	10^6 Gy	10^7 Gy	10^8 Gy	10^6 Gy	10^7 Gy	10^8 Gy
Irradiated cement compounds						
1	34.9±8.7	35.2±8.8	34.5±8.6	36.6±9.2	37.1±9.3	35.4±8.9
2	34.6±8.7	36.3±9.1	35.1±8.8	35.9±9.0	37.4±9.4	36.6±9.2
3	29.5±7.4	28.0±7.0	29.0±7.3	30.6±7.7	31.2±7.8	31.9±8.0
4	29.3±7.3	29.7±7.4	28.7±7.2	29.8±7.5	31.8±8.0	31.7±7.9
During frost resistance testing after irradiation						
1	32.5±8.1	33.2±8.3	33.4±8.4	34.5±8.6	35.0±8.8	34.4±8.6
2	31.8±8.0	33.5±8.4	33.0±8.3	33.8±8.5	34.4±8.6	33.3±8.3
3	27.4±6.9	28.8±7.2	28.7±7.2	29.1±7.3	29.3±7.3	29.9±7.5
4	26.7±6.7	28.5±7.1	28.4±7.1	29.4±7.4	29.7±7.4	29.3±7.3
During water resistance testing after irradiation						
1	35.1±8.8	34.1±8.5	35.4±8.9	33.2±8.3	33.5±8.4	34.4±8.6
2	35.2±8.8	34.5±8.6	35.3±8.8	32.6±8.2	33.3±8.3	34.1±8.5
3	30.3±7.6	29.6±7.4	31.1±7.8	28.5±7.1	29.0±7.3	30.5±7.6
4	29.7±7.4	28.5±7.1	30.9±7.7	28.6±7.2	28.3±7.1	30.4±7.6

Table 4 demonstrates that radiation exposure associated with gamma-quanta and electrons did not cause a compressive strength loss below the established limits and exceeding the established discrepancy of 20%.

These findings demonstrate that the studied cement compounds do not lose their strength capacity upon their gamma-quanta and electron exposure to a dose of 10^8 Gy.

Effects of alpha radiation were investigated for compounds No. 1 and No. 3. Upon samples receipt, ^{239}Pu was introduced into the grouting fluid; the exposure time for all samples accounted for 90 days. The experiments showed that at the highest cumulated dose of alpha radiation corresponding to 10^8 Gy, the strength of the samples did not change (within the measurement error) amounting to 35.7±8.9 MPa and 26.9±6.7 MPa for compositions No. 1 and No. 3, respectively.

Apparently, there are no fundamental differences in the radiation effects resulting from different sources of ionizing radiation, at least up to an exposure dose of 10^8 Gy.

Samples of cement compounds (compositions No. 5 and No. 6) involving LRW were studied to measure their mechanical strength, chemical resistance and resistance to radiation exposure. To accumulate a dose corresponding to 10^8 Gy, the samples were exposed to external gamma and electron irradiation. Table 5 presents the results of the cement compound testing.

Table 5. Mechanical compressive strength of cement compounds involving LRW during tests focused on frost and water resistance before and after their irradiation

Composition	Dose, Gy	Mechanical strength of the compound, MPa		
		Hardened under air-wet conditions	Following 30 freezing/thawing cycles	After being held in water for 90 days
5	0	35.8±9.0	33.4±8.4	31.0±7.8
	10^8 (electrons)	36.7±9.2	32.2±8.1	29.7±7.4
	10^8 (gamma)	34.9±8.7	33.1±8.3	28.7±7.2
6	0	34.3±8.6	32.5±8.1	29.2±7.3
	10^8 (electrons)	33.9±8.5	31.6±7.9	30.1±7.5
	10^8 (gamma)	32.6±8.2	31.1±7.8	28.8±7.2

Table 6 presents the results of ^{137}Cs, ^{90}Sr and ^{239}Pu leaching tests with irradiated samples.

Table 6. Leaching rate of ^{137}Cs, ^{90}Sr and ^{239}Pu from irradiated samples on the 28th day

Composition	Exposure dose, Gy	Leaching rate, g/cm^2·day		
		^{137}Cs	^{90}Sr	^{239}Pu
1	0	$2.2 \cdot 10^{-4}$	$9.7 \cdot 10^{-6}$	$3.1 \cdot 10^{-6}$
	10^8 (electrons)	$7.0 \cdot 10^{-5}$	$8.2 \cdot 10^{-6}$	$1.2 \cdot 10^{-6}$
	10^8 (gamma)	$9.1 \cdot 10^{-5}$	$4.3 \cdot 10^{-6}$	$8.5 \cdot 10^{-7}$
2	0	$7.1 \cdot 10^{-5}$	$4.5 \cdot 10^{-6}$	$5.8 \cdot 10^{-6}$
	10^8 (electrons)	$4.4 \cdot 10^{-5}$	$2.5 \cdot 10^{-6}$	$8.6 \cdot 10^{-6}$
	10^8 (gamma)	$2.9 \cdot 10^{-5}$	$1.3 \cdot 10^{-6}$	$1.2 \cdot 10^{-7}$
3	0	$9.0 \cdot 10^{-5}$	$1.2 \cdot 10^{-5}$	$1.4 \cdot 10^{-6}$
	10^8 (electrons)	$7.9 \cdot 10^{-5}$	$8.8 \cdot 10^{-6}$	$1.5 \cdot 10^{-6}$
	10^8 (gamma)	$9.1 \cdot 10^{-5}$	$9.5 \cdot 10^{-6}$	$9.8 \cdot 10^{-7}$
4	0	$8.1 \cdot 10^{-5}$	$9.4 \cdot 10^{-6}$	$1.1 \cdot 10^{-6}$
	10^8 (electrons)	$7.2 \cdot 10^{-5}$	$6.8 \cdot 10^{-6}$	$8.9 \cdot 10^{-7}$
	10^8 (gamma)	$6.8 \cdot 10^{-5}$	$7.9 \cdot 10^{-6}$	$7.5 \cdot 10^{-7}$
5	0	$9.0 \cdot 10^{-5}$	$1.2 \cdot 10^{-5}$	$1.4 \cdot 10^{-6}$
	10^8 (electrons)	$7.9 \cdot 10^{-5}$	$8.8 \cdot 10^{-6}$	$1.5 \cdot 10^{-6}$
	10^8 (gamma)	$9.1 \cdot 10^{-5}$	$9.5 \cdot 10^{-6}$	$9.8 \cdot 10^{-7}$
6	0	$8.1 \cdot 10^{-5}$	$9.4 \cdot 10^{-6}$	$1.1 \cdot 10^{-6}$
	10^8 (electrons)	$7.2 \cdot 10^{-5}$	$6.8 \cdot 10^{-6}$	$8.9 \cdot 10^{-7}$
	10^8 (gamma)	$6.8 \cdot 10^{-5}$	$7.9 \cdot 10^{-6}$	$7.5 \cdot 10^{-7}$

It can be noted that ^{137}Cs, ^{90}Sr and ^{239}Pu leaching rate slightly decreases with an increase in the exposure dose and continues to decrease following the introduction of bentonite clay powder. No dependence between the exposure type and the leaching rate from the studied cement compounds could be identified. For all sample compositions, the leaching rates do not exceed the regulatory limits specified for cemented RW.

Conclusions

The research performed shows that cement compounds with HLW simulator and genuine intermediate-level LRW retain their physical and mechanical characteristics both under thermal loads (up to a temperature of 90 °C) during sample solidification and dose loads of up to 10^8 Gy under long-term water exposure and alternate freezing/thawing cycles. An increase in the ambient temperature (up to 90 °C) accelerates cement hardening at its early stage, provided that the required humidity is maintained in the thermostat. Radionuclide leaching rate can be reduced to up to 30 % by introducing some bentonite clay (5 wt. %). Moreover, maximum leaching rate for almost all samples, including the irradiated ones, did not exceed 10^{-4} g/(cm^2·day) for cesium, 10^{-5} g/(cm^2·day) for strontium, and 10^{-5} g/(cm^2·day) for plutonium. An increase in the cement hardening temperature did not produce any significant effect on the radionuclides leaching rate.

Thus, the test of Portland cement-based compounds being mixed either with water or simulated HLW and intermediate-level LRW provide no evidence obstructing their consideration as a suitable matrix for HLW immobilization.

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