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Conditioning of boron-containing LRW with composite binders

Abstract. Modern management of liquid radioactive waste (LRW) requires mandatory conditioning, which they practically carried out by cementing, most often with Portland cement. As an alternative to cementing, alkaline binders are used, which are chemically more stable, increase strength over time, and do not pollute the atmosphere with CO2. Most often, the basis of such binders is granulated blast furnace slag in combination with alkaline substances. As a development of LRW management, the direction of conditioning with composite binders we formed, which combines Portland cement and blast furnace slag in composition. Composite binders can take into account the positive properties inherent in the constituent components, while the properties will depend both on the physicochemical parameters and on the conditions under which the LRW is conditioned. The work shows that boron-containing LRWs with a pH > 12 and a total salt content of 60.2% can be conditioned with composite binders, with a ratio of granulated blast-furnace ground slag (particle size < 80 μm): PC-500 cement as 1:1 with a compressive strength > 3 MPa at a temperature of 55 - 60°C. The amount of LRW (imitate) in the compound can be 35 - 40%. Alkaline substances included in the LRW composition they used as a binder hardening activator, without the addition of additional alkalis and liquid glass. Depending on the types of components involved: Portland cement, blast-furnace slag or composite binder, samples of compounds with a compressive strength of mainly > 10.3 MPa we obtained.

Keywords: boron-containing LRW, conditioning, composite binder

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Introduction.

Conditioning of radioactive waste, i.e. converting it into a form suitable for transportation, disposal and long-term storage, is an important area of safe operation of nuclear power. As this area develops, new approaches and new materials for conditioning appear, in particular the use of composite binders.

Composite binders we understood as binders that consist of several components with different mechanisms of compound formation. The advantage of composite binders is that due to the consistency between the conditioning mechanisms, there is a possibility of their synergistic use.

In the work, Portland cement and blast furnace granulated slag we considered as components of the composite binder.

It they known that the most common method of conditioning RRW is their cementation. Most often, Portland cement with a small admixture of bentonite clay in a dispersed state we used for this. The resulting compounds or characterized by high strength and meet the requirements for cemented waste. Currently, the search for a replacement for Portland cement is increasingly expanding. This is because during the production of 1 ton of cement, more than 800 kg of CO_2 they emitted into the air, which in conditions of global warming contributes to its spread. In addition, in conditions of combining Portland cement with boron-containing salts of salt melts, there is uncertainty about the preservation of sufficient strength of Portland cement compounds for a long time.

As an alternative direction, cementation of waste with alkali-activated cements, which are formed from natural and synthetic calcium aluminosilicates under alkaline conditions, is being developed [1 – 8].

The combination of Portland cement with the component of alkaline cement - slag has certain features.

Literary review and problem statement.

If the water in the waste during the conditioning of Portland cement is part of the calcium hydrosilicates and hydroaluminosilicates and remains in a bound state, then the water of alkaline cements performs the functions of a transport medium and evaporates upon completion of the synthesis. When trying to create compounds based on alkaline cements that are sufficiently strong, their porosity should be less than 0.5%. With such porosity, evaporation can be very long, and in this case, if there is a large amount of free water in the samples, part of it from the composite binders we be taken by Portland cement.

The mechanisms of immobilization of radionuclides by Portland cement and alkaline cement components are different. As for the immobilization of radionuclides by Portland cement binders, it is impossible in confidently state the mechanism of immobilization based on the negligible mass concentrations of radionuclides. This may be sorption on active centers of mineral particles, or in the form of complexes such as $Cs[Al(OH)_4]$, $CsAl(SiO_2)_2$, etc. The adequacy of the immobilization mechanism at a negligible low level to processes recorded at the macro level is still subject to research.

It has been established that at the beginning of hardening, the hydration products of alkaline cement are, regardless of the basicity of the slag and the type of alkaline component, as a rule, low-basic calcium hydrosilicates of the type CSH(B), CSH_2, $C_5S_6H_3$. These hydrated new formations are mainly in the X-ray amorphous state, as well as complex gel-like compounds of alkali and alkaline-earth aluminosilicate composition [3].

Later, crystallization of calcium hydrosilicates occurs in the system, which begins earlier than the complex alkali and alkaline-earth hydroaluminosilicates crystallize. Over time, analcime $NaAlSi_2O_6{\cdot}H_2O$ (d/n =0.56; 0.40; 0.34; 0.25; 0.23; 0.17 nm), mixed sodium-calcium hydrosilicates of the type $Na_2Ca_2Si_3O_8{\cdot}OH$ (d/n =0.7; 0.40; 0.39; 0.33; 0.27; 0.175 nm) and other products are formed, which play an important role in the genesis of microstructures and the strength of the binder [3].

Alkaline cement matrices can retain radionuclides 137Cs, 90Sr, and hazardous substances in their volume for a long time due to sorption and chemical binding [5, 7, 8].

In the Portland cement component of the composite binder, free boron forms insoluble compounds of the type $CaB_2O_4{\cdot}4H_2O$ and $Ca(H_2BO_3)_2{\cdot}4H_2O$. Boron anions BO_3^{3-} and BO_4^{5-} slow down the hydration of C_3A and C_3S due to the low solubility of calcium ions in the solution [9 – 12].

In the alkaline cement component of the composite binder, free boron atoms in an alkaline environment in the presence of Ca cations can form compounds $CaO{\cdot}B_2O_3{\cdot}6H_2O$ and $2CaO{\cdot}3B_2O_3{\cdot}18H_2O$. Due to the formation of the N–B–A–S–H gel (hydrated sodium bora aluminosilicate), which promotes the sorption-chemical binding of radioactive cations of cesium (Cs^+), barium (Ba^{2+}), and strontium (Sr^{2+}) in the frameworks of boron-containing aluminosilicates [13 – 16].

The results of the practical application of a composite binder (slag Portland cement) for conditioning waste from Khmelnytskyi and Chernobyl NPPs we given in [3, 5, 7]. Regarding the waste, this indicated that the salt content was 453 g and 365 g, respectively. At the same time, Na^+ cations were 1.4 times more than BO_3^- in the case of Khmelnytskyi NPP waste, borates in Chernobyl NPP waste were absent, and K+ cations were also absent, and the waste included a significant amount of nitrates. The binder contained 50% clinker and 50% technogenic sludge. The resulting compounds had a compressive strength of 11 MPa for Khmelnytskyi NPP waste and 6.2 MPa for Chernobyl NPP waste. The salt content in the samples was 28.6% and 21.1%, respectively. Positive results we obtained for leaching. The indicator of $1{\cdot}10^{-3}$ g/cm²·day we observed already on the third day, while when using Portland cement after 3 months. Unfortunately, it is difficult to repeat the above experiments when conditioning LRW of Zaporizhzhia NPP, since neither the physicochemical properties of Portland cement (clinker) and technogenic waste nor the conditions for manufacturing samples they given.

From the above, the goal of the work follows: to obtain samples in which the boron-containing imitation of LRW they conditioned by a composite binder or its components with the study of their basic properties.

Materials and methods.

To perform the work, a composite binder slag Portland cement of the brand SPC III/A - 400 (SEM III) according to DSTU BV.2.7-46:2010 was used, which includes Portland cement PC I-500R-N, produced by PrJSC "Ivano-Frankivsk Cement" and granulated slag of the Kamiansky Metallurgical Plant, the chemical composition of which is presented in Table 1.

Table 1 – Chemical composition of the components of the composite binder

Oxides/substances	Cement,%	Slag, %
SiO_2	21,9	41,6
Al_2O_3	5,42	4,9
Fe_2O_3	3,61	0,42
CaO	61,6	45,6
MgO	0,87	3,84
TiO_2	-	1,1
SO_3	2,6	-
H_2S	-	0,1
R_2O	1,1	-
Cl	0,02	-
LDC	2,2	0,72

Mineralogical composition of cement clinker, %: C_3S - 60.5; C_3A - 6.7; C_2S - 14.9; C_4AF - 12.4. Slag parameters: specific surface area 303 m^2/k; density, 2.82 g/cm^3. When combining Portland cement and slag in the above-mentioned proportion of 50%: 50%, the following results were obtained: the beginning of hardening of the mixture was 130 min.; compressive strength, MPa: 7 days – 24.9; 28 days – 44.7.

According to the chemical analysis of the bottom residue of the Zaporizhzhia NPP with a salt content of 800 g/cm3 [8], an imitation of the RRB was made with a similar composition, wt%: $Na_2B_4O_7{\cdot}5H_2O$ – 36.1; $NaNO_3$ – 7.8; NaOH – 13.0; KOH – 2.1; NaCl – 0.3; Fe_2O_3 – 0.3; H2O – 39.8. The imitation also included $SrCl_2{\cdot}6H_2O$, $CsNO_3$ and surfactants, which in total amounted to 0.5%. The density of the imitation was 1.54 g/cm^3, the total salt content was 60.2%.

When forming compounds 3x3x3 cm (Fig.1), in addition to the imitation, active mineral additives we introduced into the composition of the composite cement - bentonite, metakaolin, zeolite, mechanically activated slag in a vibration mill.

Figure 1 – Compound samples 3x3x3 cm in shape

Sample 128, which used Portland cement and bentonite, is noteworthy. The sample they characterized by a high compressive strength (> 28 MPa) and low porosity (≈ 4%). For the Portland cement used, it is satisfactory, but their possible change over time can only be established was result of long-term experiments.

Sample 126, in which the market cement of the SPC IIIA/400 brand we used as a composite binder, which contains 50% Portland cement PC – 500 and 50% ground granulated blast furnace slag, had sufficient strength (13.9 MPa), porosity was 2.4%.

As the initial parameters characterizing the properties of the compounds, density, porosity and compressive strength we taken. These parameters were determined according to the current technical specifications of Ukraine. The criterion chosen was the comparison of the strength at the branded age with the strength according to DSTU B V.2.7-214:2009, which for such materials should be not less than 4.5 MPa.

Results and their discussion.

An important indicator in the conditioning of LRW with composite binders is the temperature of the process itself. It has been established that at a temperature of 55°C – 60°C, the salt melt imitation is evenly mixed with the composite binder powder and forms a pasty mixture with a plastic strength of about 2 kPa – 3 kPa.

Such a mixture can contain (36÷38) % of the imitation and has sufficient plasticity to fill the corresponding forms for the purpose of subsequent hardening and gradual increase in strength.

For the experiments, samples we manufactured and tested, in which the same imitation we used, and the hardening we activated by alkalis included in the imitation [17]. The results of the studies they given in Table. 2.

Table 2 - Comparative composition of compounds, % and their properties

Sample numbers / Components	126	127	128	129	130	131	132
PC-500	-	-	56.6	-	-	-	53.6
SPC III A/400	56.6	-	-	-	54.5	54.5	-
Slag		56.6	-	-	-	-	-
Bentonite	5.7	5.7	5.7	5.6	-	-	-
Metakaolin	-	-	-	-	5.4	-	-
Zeolite	-	-	-	-	-	5.5	5.4
Imitate	37.7	37.7	37.7	37.0	36.4	36.4	35.7
Water	-	-	-	2.0	3,6	3.6	5.4
Mechanically activated slag	-	-	-	55,6	-	-	-
Compressive strength, MPa at 28 days	13.9	11.2	28.0	8.4	12.0	13.5	14.0
Density, g/cm^3	1.91	1.81	2.1	1.81	1.94	1.84	1.91
Porosity, %	2.4	8.9	4.1	11.6	24.5	11.1	4.2

If only one slag is used as a binder (sample 127), we will obtain a decrease in the compressive strength to 11 MPa and an increase in porosity to almost 9%. This is the cheapest way to condition the LRW due to the low cost of granulated slag. It requires additional research into the physicochemical properties of the slag, conditioning conditions, and other quality indicators of the compounds - frost resistance, water resistance, leaching, etc.

They known that when cooling the salt melt, sodium tetraborate contained in its composition we converted into sodium metaborate. The latter crystallizes in the volume of the melt in the form of crystals of various sizes. However, when mixing composite binders containing dispersed particles of the crystalline phase of the slag, and free boron can form compounds, which we discussed above, the addition of bentonite or zeolite powder is not mandatory, it only increases the plasticity and sorption properties of the compounds. The addition of ground zeolite instead of ground bentonite clay did not change this indicator (sample 131), but increased the porosity.

Metakaolin in the compounds did not increase their strength, but significantly increased the porosity of the samples to 24.5% (sample 130). In this sample, the composite cement turned out to be a satisfactory binder for conditioning the LRW.

The porosity of the sample with mechanically activated slag (activation at 16,000 rpm for 20 min) sample 129 increased. The reasons for the increase in porosity are subject to further study.

The results of conditioning the LRW with Portland cement combined with zeolite (sample 132) we obtained. The compounds have a strength of 14 MPa and low porosity [18].

Conclusions.

A series of experiments we made and conducted, in which the same imitation of RRB we conditioned with different binders: Portland cement, slag Portland cement (composite binder) and granulated slag. It we shown that the composite binder is suitable for conditioning boron-containing LRW in an amount by weight of up to 35%. Conditioning they carried out at a temperature of (55 - 60) °C. Activation of slag hardening in the composition of the composite binder they carried out by alkalis contained in the LRW. Optimization of the composition of the compounds can be base on the results obtained: the more Portland cement in the composite binder, the higher the strength of the samples, but with a decrease in slag in the samples, a decrease in their chemical resistance should be expecte.

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Кондиціонування борвмісних РРВ композиційними зв'язуючими

Аннотація. Сучасне поводження з рідкими радіоактивними відходами (РРВ) передбачає обов'язкове їх кондиціонування, що практично виконується шляхом цементування найчастіше портланцементом. Як альтернатива для цементування залучаються лужні в'яжучі речовини, які хімічно більш стійкі, збільшують міцність з часом, не забруднюють атмосферу СО2. Найчастіше основою таких зв'язуючих є гранульований доменний шлак у поєднанні з лужними речовинами. Як розвиток поводження з РРВ формується напрямок кондиціонування композиційними зв'язуючими, що поєднує у складі портландцемент та доменний шлак. Композиційні зв'язуючі можуть враховувати позитивні властивості притаманні складовим компонентам, при цьому властивості залежатимуть як від фізико-хімічних показників так і від умов, за якими відбувається кондиціонуванні РРВ. В роботі показано, що борвмісні РРВ, які мають pH > 12 при загальному вмісті солей 60,2 % можуть бути кондиціоновані композиційними зв'язуючими, при співвідношенні гранульований доменний мелений шлак (розмір частинок < 80 мкм): цемент ПЦ-500 як 1:1 з міцністю при стсику > 3 МПа при температурі 55 – 60□С. Кількість РРВ (імітату) у компаунді може становити 35 – 40 %. У якості активатора твердіння зв'язуючого можуть бути використані лужні речовини, які входять до складу РРВ, без внесення додаткових лугів та рідкого скла. В залежності від видів залучених компонентів: портландцемент, доменний шлак чи композиційне зв'язуюче отримані зразки компаундів з межею міцності на стиск переважно >10,3 МПа.

Ключові слова: борвмісні РРВ, кондицiювання, композиційне зв'язуюче.

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