

Було розроблено склади матриць для утилізації аніонно-обмінних смол з низьким рівнем радіоактивності, що утворюються внаслідок роботи атомних реакторів, з використанням гібридних лужних цементів. Показано результати дослідження експлуатаційних властивостей кінцевого продукту (міцності, вилуговання, радіаційної стійкості, тощо) та доведено, що за своїми фізико-механічними та фізичними властивостями розроблені склади відповідають вимогам стандартів КНР GB 7023 та GB 14569

**Ключові слова:** цементування, радіоактивні відходи низького рівня активності, іммобілізація, аніонно-активні смоли

Были разработаны составы матриц для утилизации анионно-обменных смол с низким уровнем радиоактивности, которые образуются как следствие работы атомных реакторов, с использованием гибридных щелочных цементов. Показаны результаты исследований эксплуатационных свойств конечного продукта (прочности, радиационной стойкости и пр.) и доказано, что по своим физико-механическим и физическим характеристикам разработанные составы соответствуют требованиям стандартов КНР GB 7023 и GB 14569

**Ключевые слова:** цементирование, радиоактивные отходы низкого уровня активности, иммобилизация, анионно-активные смолы

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# EFFICIENCY OF ALKALI ACTIVATED HYBRID CEMENTS FOR IMMOBILIZATION OF LOW-LEVEL RADIOACTIVE ANION-EXCHANGE RESINS

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## 1. Introduction

Cementation is one of the most widely used methods of immobilization of low-level radioactive wastes [1]. A great variety of low-level radioactive wastes requires a proper choice of method for preparation of a cement-based grout, for which an optimal cement composition should be chosen meeting to the highest extent the requirements with regard to long-term disposal of these solidified waste forms. Traditional OPC compositions are not able to provide all necessary service properties of the final product so it is necessary to offer new, more effective binder for such systems.

The taken direction is actual because of wide distribution of nuclear power plants and reactors all over the world and high environmental risks at the stage of storing wastes from these reactors.

## 2. Literature review and problem statement

One of the major requirements is safety of binding such elements as  $^{137}\text{Cs}$  and  $^{90}\text{Sr}$ . Cesium is the most difficult to

process element because it is poorly bound within hydration products of cementitious/binding agents based on calcium silicates and aluminates [2]. However, as it follows from the publications [3, 4] it can be chemically incorporated into aluminosilicate and other zeolite-like structures, which are known to be major structure forming phases of the alkali activated cements [5–7]. The efficiency of the alkali activated cements for immobilization of low-level radioactive wastes is supported by a number of researchers. Thus, in [8, 9] the principal possibility of using alkali activated cements for immobilization of radioactive materials was shown. Papers [9–11] show effectiveness of alkali activated cements application because of incorporation of radioactive components into the composition of cement new formations. In this case, immobilization is going not only in a physical way, but also in chemical. Results of tests of different service properties of alkali activated matrixes show [12–14] that application of that binder is very effective from the point of view of compressive strength and leaching behavior.

Synthesis of the zeolite-like hydration products [16–18] with rather high sorption ability in the alkali activated cement stone is an additional factor to fix physically the

incorporated radioactive elements. Thus, radionuclides not only participate in the cement stone structure formation but are locked in a three-dimensional lattice of a zeolite-like matrix with high energy – non-saturated cavities (Fig. 1).

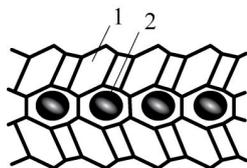


Fig. 1. Ecologically dangerous elements packed in the synthetic zeolite-like matrix of the alkali activated cements: 1 – matrix; 2 – elements

Among many other low-level radioactive wastes the ion-exchange resins are very difficult to process by cementation, in particular, the most difficult are anion-exchange resins (pH<5) [19, 20].

A complexity of their immobilization is, that in the medium of the hydrating cement with high pH-values a generation process of these resins, caused by their structural peculiarities proceeds faster (Fig. 2), resulting in destruction of the cement stone with loss of its water resistance. That is why such cement matrix should be chosen for immobilization (solidification) in which the pH-value of the medium during hydration and hardening would be lowering continuously, retarding a regeneration process, and would not affect physical-mechanical properties of the resulted product (final product or solidified waste form).

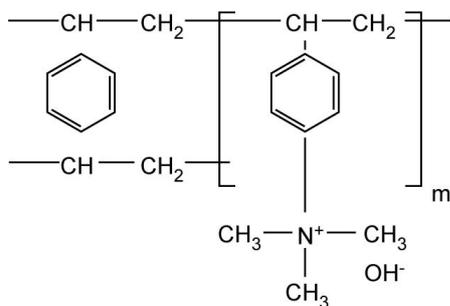


Fig. 2. Chemical formula of anion-exchange resin

According to the earlier studies [21–23], hybrid alkali activated cements (AAC) are among the best binding agents to form a cement matrix with target properties.

### 3. The purpose and objectives of the study

The purpose of the work was to substantiate a choice of AAC composition and to study the properties of the hybrid AAC-based grouts and solidified waste forms which contain anion-exchange resins.

To realize these goals, the following tasks were formulated:

- to obtain effective matrixes for solidification of low-level radioactive anion exchange resin;
- to find out regularities of influence of different constituents of the cement on service properties;
- to develop the final product composition able to be used according to the PRC standards.

### 4. Materials and testing methods

The pulp of the following composition: anion-exchange resin with water content of the pulp =50 % by mass and CsNO<sub>3</sub> – 1.5 g/l was used in testing.

After mixing and storage for 7 days (recommendation of the nuclear power plant) the pulp density was 1070 kg/m<sup>3</sup> and its pH=5. The hybrid AAC-based grouts were prepared in a Hobart mixer. The hybrid AAC-based grouts should meet the following requirements (specified by the nuclear power plant): waste (pulp) loading – at least 60 % by volume, flowability ≥180 mm, initial setting time ≥2 hr, final setting time <36 hr.

The requirements for the grouts are as follows: waste (pulp) loading – not less than 60 % by volume, flow ≥180 mm, initial setting time ≥2 hours, final setting time <36 hours. The alkali-activated cement-based grouts have been prepared in a standard mixer for cement mortar (“Hobart”, Australia).

First, the pulp was mixed in a plastic container where it was kept for homogeneous distribution of the resin particles. The properly homogenized ion-exchange resin pulp was brought into a mixer bowl, then the alkali activated cement components were added and a final mixing took place (for 7 min).

A flowability of the alkali activated cement-based grouts was measured using two methods: the first – a Vicat cone (standard equipment for testing cement, produced in the USSR) and with the help of a special testing equipment (a paddle with openings, specified by PRC standard, produced in China). Testing by the latter method showed that in case the flow value of 180 mm the “clarification” of the openings took place 1–2 seconds after the paddle was lifted from the mixer bowl.

To study if the AAC-based grouts are in compliance with the P. R. China standards GB 7023 and GB 14569 requirements the beam (40×40×160 mm) and cylinder (d=h=50 mm) specimens have been prepared.

### 5. Results and discussions

To substantiate a choice of hybrid alkali activated cement composition, which would allow to achieve target properties a role of each cement component, namely: composition and content of alkaline components, type of calcium-containing constituent, type of clay were studied.

Alkaline compounds play an important role in providing flow of processes of hydration and hardening of the AAC-based grouts. With time the alkaline compounds tend to be bound with the formation of insoluble phases of the Na<sub>2</sub>O–Al<sub>2</sub>O<sub>3</sub>–SiO<sub>2</sub>–H<sub>2</sub>O type, thus reducing pH-values of the hybrid AAC stone. However, with the increase in alkali content no optimal lowering of the pH took place and since the processed resin is an anion-exchange resin, the process of its regeneration in the solidified cement stone began resulting in its destruction. This coincides well with the data given in Table 1 (composition 7F-2).

As it follows from these data, the increase in alkali content in these compositions results in quick strength gain at early stages of hardening, however, at the later stages the grouts tend to less lose their water resistance.

Also, the specimens of the composition 7F-2, with 5.8 % Na<sub>2</sub>CO<sub>3</sub> by mass turned out to be not water resistant. The

specimens of the compositions F-5 and 7F-0 with 1.7 and 2.7 % Na<sub>2</sub>CO<sub>3</sub> by mass respectively were found to be optimal ones with regard to water resistance and strength development. No doubt that the composition F-5 with 1.4 % Na<sub>2</sub>CO<sub>3</sub> by mass is the best from the point of view of deteriorating aggressive action of the anion exchange resin, however, the strength of the specimens was insufficient.

Table 1

The influence of alkaline component content

Composition	Composition number				
	F-6	F-5	7F-0	7F-2	F-0-P-1
	%				
Slag (ggs)	36.4	34.1	37.2	34.4	35.3
Na <sub>2</sub> CO <sub>3</sub>	–	1.4	2.7	5.8	2.5
Bentonite	4.7	4.7	5.0	5.1	4.7
OPC	8.2	8.2	8.7	8.9	8.2
Resin pulp	39.8	40.0	41.8	41.0	39.7+3.1 (NaOH)
H <sub>2</sub> O	11.9	11.6	4.6	4.8	6.5
Flow, mm	165.0	158.0	140.0	160.0	180.0
RAW loading, % by volume	56.7	56.9	62.7	61.7	59.2
Compressive strength (age – 7 days), MPa	4.6	5.0	9.4	9.7	–
Compressive strength (age – 28 days), MPa	4.0 (13 d)	5.3 (18 d)	8.4 (18 d)	10.1 (18 d)	8.8 (18 d)
Water resistance	+	+	+	+(-)	–

Note: “+” – water resistant specimens; “–” – not water resistant specimens

*The influence of OPC content.* In order to increase the strength of the final products containing minimal quantities of the alkaline component, the OPC in quantities varying from 8.2 to 21.8 % by mass was added to the hybrid AAC-based grout compositions (Table 2).

Table 2

The influence of OPC content

Composition	Composition number			
	F-5	F-14	F-C-1	F-C-2
	%			
Slag (ggs)	34.1	34.6	30.5	21.8
Na <sub>2</sub> CO <sub>3</sub>	1.4	1.6	1.6	1.5
Bentonite	4.7	5.2	5.3	4.9
OPC	8.2	11.5	16.4	21.8
Resin pulp	40.0	39.4	44.0	41.1
H <sub>2</sub> O	11.6	7.7	2.2	8.9
Flow, mm	158.0	175.0	170.0	160.0
RAW loading, % by volume	56.9	59.0	66.7	60.0
Compressive strength (age – 7 days), MPa	3.9	–	5.9	7.75
Compressive strength (age – 28 days), MPa	–	4.8 (5 days)	–	–
Water resistance	–	+	+	+

Note: “+” – water resistant specimens; “–” – not water resistant specimens

As it follows from Table 2, with the increase of the OPC content the strength of the specimens tends to increase from 3.9 to 7.75 MPa after 7 days of hardening. It is important that water resistance is achieved only starting from the OPC content of 11.5 % by mass (the composition F-14), after 7 days of hardening. Too large quantities of the OPC additive, though makes the strength development more intensive especially at the early ages, however will negatively affect an immobilizing capability of the cement stone, since the lower content of granulated blast-furnace slag the lower content of the CSH-phase in the hydration products.

*The influence of alkaline component content.* With taking into account the earlier described results the influence of a combined action of the alkaline component and OPC was studied. The results of tests are given in Table 3. As it follows from the table, with the contents of the alkaline component varying 1.2...2.0 % by mass and contents of OPC varying from 11.5 up to 23.2 % by mass all test specimens after 3 days were water resistant, and after 5 days their strength varied from 2.8 to 6.1 MPa.

Table 3

The influence of alkaline component content

Composition	Composition number					
	F-11	F-11K	F10	F13	F14	F-12K
	%					
Slag (ggs)	30.0	30.4	30.2	23.3	34.6	29.4
Na <sub>2</sub> CO <sub>3</sub>	1.6	1.7	1.2	1.2	1.6	2.0
Bentonite	5.2	–	5.2	5.2	5.2	–
Kaolin	–	5.4	–	–	–	5.1
OPC	16.0	16.4	16.0	23.2	11.5	15.8
Resin pulp	39.5	41.9	39.0	39.4	39.4	41.9
H <sub>2</sub> O	7.7	4.2	7.7	7.7	7.7	4.2
Flow, mm	173	190	150	140	175	155
RAW loading, % by volume	59.0	63.4	59.0	59.0	59.0	63.5
Compressive strength (age – 7 days), MPa	6.12 (5d)	6.4 (3d)	2.8 (5d)	6.1 (5d)	4.8 (5d)	6.25 (3d)
Water resistance	+	+	+	+	+	+

With taking the data on the lower water resistance of the specimens with the increased contents of the alkaline component into consideration the compositions F-10, F-11, F-13 and F-14 have been chosen for further investigation.

A conclusion was drawn that the OPC content is a more sensitive factor affecting the consistency of the hybrid AAC-based grouts.

*The influence of clay component.* A type of clay component (kaolin or bentonite) is known to affect both properties of grouts (flowability, setting times, etc.) and those of final products (waste loading, water resistance, strength, corrosion resistance, freeze-thaw resistance, leachability etc.). To study this influence at a stage of composition design the compositions F-11 and F-12, which vary in content of the alkaline component – 1.6 and 2.0 % by mass and in which the bentonite was replaced with kaolin have been chosen.

The results of tests are given in Table 4. As it follows from the table, a replacement of the bentonite with kaolin positively affected both strength properties of the grouts and final products. By changing the contents and type of clay component a possibility occurs to considerably affect the consistency of the grouts.

Analyzing the obtained results, the following compositions have been chosen to further study properties of the solidified waste forms, these were: the compositions F-10, F-11, F-11K, F-12K and F-14.

Table 4

The influence of clay component

Composition	Grout composition			
	F-11	F-11K	F-12	F-12K
	%			
Slag (ggbfs)	30.0	30.4	30.2	30.2
Na <sub>2</sub> CO <sub>3</sub>	1.6	1.7	2.1	2.0
Bentonite	5.2	–	5.3	–
Kaolin	–	5.4	–	5.3
OPC	16.0	16.4	16.3	16.3
Resin pulp	39.5	41.9	4.0	40.0
H <sub>2</sub> O	7.7	4.2	6.2	6.2
Flow, mm	173.0	190.0	150.0	155.0
RAW loading, % by volume	59.0	63.4	59.0	60.0
Compressive strength (age – 28 days), MPa	6.12 (5 d)	6.4 (3 d)	2.8 (5 d)	6.25 (3 d)
Water resistance	+	+	+	+

Note: “+” – water resistant specimens; “–” – not water resistant specimens

Leaching behavior of Cs is shown in Table 5. The results obtained with all compositions under study are superior to target requirement ( $1.0 \cdot 10^{-3}$  cm/d), thus testifying to a high efficiency of hybrid AAC as binding agents for immobilization of radionuclides.

Mechanical properties were studied on beam specimens  $40 \times 40 \times 160$  mm, which were allowed to harden at  $t = 20 \pm 2$  °C and  $RH \geq 90$  % and also subjected to crash after fall from 9 m height. The results given in Table 6 and in Fig. 3 show that the compositions F10, F11K, F12K and F13 had met the necessary requirements with regard to strength properties and drop tests (strength is less than 7.5 MPa).

Table 5

Leaching from final products

Grout composition	Open surface (volume) of a specimen, cm <sup>2</sup> (cm <sup>3</sup> )	Cumulative leaching of Cs from specimen in water at 42 days, g	Cs- content in a specimen, g	Cumulative leaching of Cs at 42 days (P42), cm
F-10	112.6632 (91.3583)	0.0285	0.0596	$5.53 \cdot 10^{-5}$
F-11	101.1080 (78.0698)	0.018	0.0494	$4.12 \cdot 10^{-5}$
F-11K	99.6636 (76.4087)	0.0210	0.0553	$4.27 \cdot 10^{-5}$
F-12K	101.1080 (78.0698)	0.0255	0.0526	$5.48 \cdot 10^{-5}$
F-13	102.5524 (79.7308)	0.0270	0.0511	$6.02 \cdot 10^{-5}$
F-14	105.4412 (83.053)	0.0225	0.0538	$4.70 \cdot 10^{-5}$

Table 6

Strength characteristics. Test results

Grout composition	Flexural strength, MPa	Compressive strength, MPa
F10	1.8	8.3
F11	2.2	7.2
F11K	3.1	12.5
F12K	2.3	8.9
F13	2.4	10.6
F14	1.6	5.6

No any serious changes of specimen forms after the cylinder specimens ( $d=h=50$  mm) fall from 11 m was found.

Biological resistance was evaluated by changes of flexural strength of beam specimens ( $40 \times 40 \times 160$  mm) after their storage in the H<sub>2</sub>SO<sub>4</sub> solution with pH=3 during 3 months. In parallel, the reference specimens were kept in water. As it follows from the results represented in Table 7, the only composition – the composition F14 had the coefficient of corrosion resistance below 0.8.

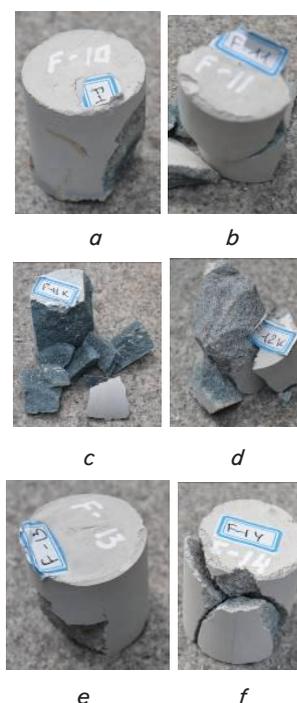


Fig. 3. Photos of specimens after drop test: a – F10; b – F11; c – F11K; d – F12K; e – F13; f – F14

Table 7

Biological stability. Test results

Grout composition	Compressive strength, MPa		Flexural strength, MPa		Coefficient of corrosion resistance
	before test	after test	before test	after test	
F10	8.3	9.1	1.8	1.6	0.89
F11	7.2	8.0	2.2	2.0	0.91
F11K	12.5	13.0	3.1	3.0	0.97
F12K	8.9	10.7	2.3	2.0	0.87
F13	10.6	14.6	2.4	2.3	0.96
F14	5.6	12.1	1.6	1.2	0.75

Fire resistance was studied on beam specimens (40×40×160 mm), which had been preliminary cured at t=60±5 °C for 20–24 hr, then heated up to t=600 °C and allowed to mature for 30 min. After cooling, the mass loss should not exceed 50 %.

Test results given in Table 8 and Fig. 4 show that all compositions under study meet the requirements for fire resistance.

Freeze-thaw resistance was studied on beam specimens (40×40×160 mm). The following test conditions were applied: t=(–) 20 °C, (+) 20 °C, freezing – 3 hr; thawing – 4 hr. After 10 freeze-thaw cycles the loss of strength is not allowed to exceed 25 %. As it follows from Table 9, all hybrid AAC-based grouts under study met the requirements for freeze-thaw resistance.

Table 8

Fire resistance. Test results

Grout composition	Mass, g		Mass loss Δm, %
	before test	after test	
F10	285	216	24.2
F11	282	212	24.8
F11K	336	231	31.2
F12K	317	217	31.5
F13	319	220	31.0
F14	298	212	28.8

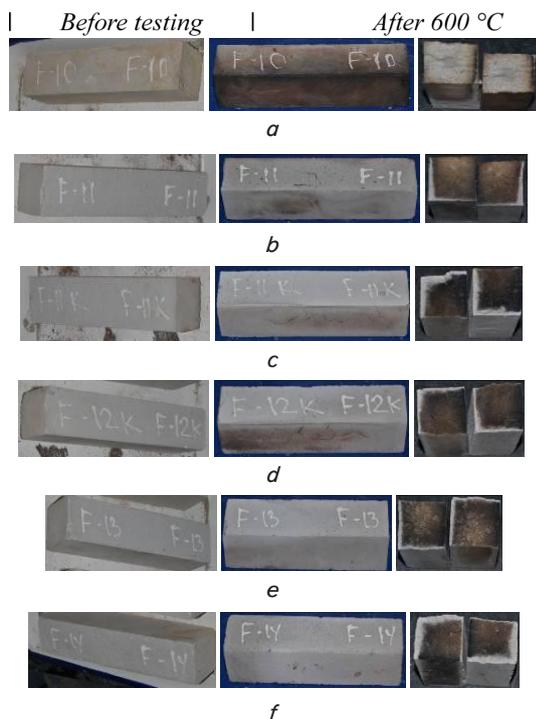


Fig. 4. Photos of specimens for testing fire resistance: a – F10; b – F11; c – F11K; d – F12K; e – F13; f – F14

Resistance in long-term storage in water is one of the most important characteristics which confirm the validity of a proper choice of AAC composition as to alkali contents. To

study this property, the beam specimens (40×40×160 mm) were kept in water at t=25±5 °C for 90 days. The strength loss was not allowed to exceed 25 %. Results of the test are given in Table 10.

Table 9

Freeze-thaw resistance. Test results

Grout composition	Compressive strength, MPa		Strength variation, %
	before test	after test	
F10	8.3	7.1	–14.5
F11	7.2	7.7	–6.5
F11K	12.5	14.1	+12.8
F12K	8.9	10.0	+12.3
F13	10.6	12.0	+13.2
F14	5.6	7.2	+28.6

Note: (+) – strength gain; (–) – strength loss

Table 10

Resistance for long-term storage in water. Test results

Grout composition	Compressive strength, MPa		Strength changes, %
	before test	after test	
F10	8.3	5.8	–30.1
F11	7.2	8.3	+15.3
F11K	12.5	10.8	–13.6
F12K	8.9	10.7	+20.2
F13	10.6	10.6	0.0
F14	5.6	6.3	+12.5

As it follows from the test results (Table 10) the only test specimens that had not passed it were the specimens of the composition F10. This can be attributed to insufficient contents of alkaline activator in this hybrid AAC composition. All other compositions had met the requirements of the standard and are able to be applied.

## 6. Conclusions

The examples of compositional build up of the hybrid alkali activated cements intended for immobilization of anion-exchange resins suggested to make the following conclusions:

- 1) the possibility of obtaining effective matrixes for immobilization of anion-active resin is proved. Anion-active resin pulp content is up to 50 % by mass of the final product;
- 2) in the hybrid AAC composition a content of the alkaline activator is the most influential factor from the point of view of stability of the solidified waste forms in time;
- 3) the Ca-containing component and clay component act chiefly as regulators of technology-related properties of the hybrid AAC-based grouts;
- 4) according to the obtained test results, the solidified waste forms made using the developed hybrid alkali activated cement compositions are superior in their basic properties to those specified in the P.R.China standards GB 7023 and GB 14569.

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