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Original Article

Evaluation of cementation of intermediate level liquid waste produced from fission ^{99}Mo production process and disposal feasibility of cement waste form

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ABSTRACT

The Korea Atomic Energy Research Institute (KAERI) is planning the construction of the KIJANG Research Reactor (KJRR) for stable supply of ^{99}Mo. The Fission ^{99}Mo Production Process (FMPP) of KJRR produces solid waste such as spent uranium cake and alumina cake, and liquid waste in the form of intermediate level liquid waste (ILLW) and low level liquid waste (LLLW). This study thus established the operating range and optimum operating conditions for the cementation of ILLW from FMPP. It also evaluated whether cement waste form samples produced under optimum operational conditions satisfy the waste acceptance criteria (WAC) of a disposal facility in Korea (Korea radioactive waste agency, KORAD). Considering economic feasibility and safety, optimum operational conditions were achieved at a w/c ratio of 0.55, and the corresponding salt content was 5.71 wt%. The cement waste form samples prepared under optimum operational conditions were found to satisfy KORAD's WAC when tested for structural stability and leachability. The results indicate that the proposed cementation conditions for the disposal of ILLW from FMMP can be effectively applied to KJRR's disposal facility.

1. Introduction

Radioisotopes play an important role in medical industries across the globe. Among the various radioisotopes, ^{99m}Tc is a short-lived nuclide widely used in diagnostic imaging and radiotherapy, and has been seeing a growing demand [1–4]. ^{99m}Tc can be obtained through a ^{99m}TC generator with molybdenum produced in a research reactor. However, ^{99}Mo for ^{99m}TC generators is supplied by aging reactor production facilities, leading to concerns over the unstable supply of ^{99}Mo [5,6]. Against this backdrop, the Korea Atomic Energy Research Institute (KAERI) is planning the construction of the KIJANG Research Reactor (KJRR) for stable ^{99}Mo supply [7].

KJRR's Fission ^{99}Mo Production Process (FMPP) uses Low Enriched Uranium (LEU), and does not re-process unprocessed residue and undissolved residue [8]. The types of waste produced by KJRR include solid spent uranium cake, solid alumina residue (Al_2O_3 cake), intermediate level liquid waste (ILLW), and low level liquid waste (LLLW) [9]. The solution that first passes the alumina column for separation of ^{99}Mo is ILLW, and the solution that undergoes ion exchange and adsorption becomes ILLW. Fig. 1 shows the FMPP of KJRR, and the types of radioactive wastes produced at each stage.

This study examines the processing of ILLW among various types of waste generated by KJRR's FMPP. The International Atomic Energy Agency (IAEA) has reported on processing methods employed for ILLW disposal by countries with ^{99}Mo production facilities [10]. Canada solidifies wastes into cement or bitumen depending on chemical composition and radioactive components. Belgium mixes waste in the slurry state with molten bitumen, and solidifies solid particles remaining after evaporation with bitumen. South Africa, Australia, and the Netherlands have chosen cementation for the disposal of ILLW. Solidified waste must satisfy the waste acceptance criteria (WAC) of radioactive waste disposal facilities. In Belgium, waste is stored in accordance with the IAEA Code of Practice 50-C-QA. Other countries have not provided details of storage. The report also lacked details on processing methods and acceptance criteria of disposal facilities.

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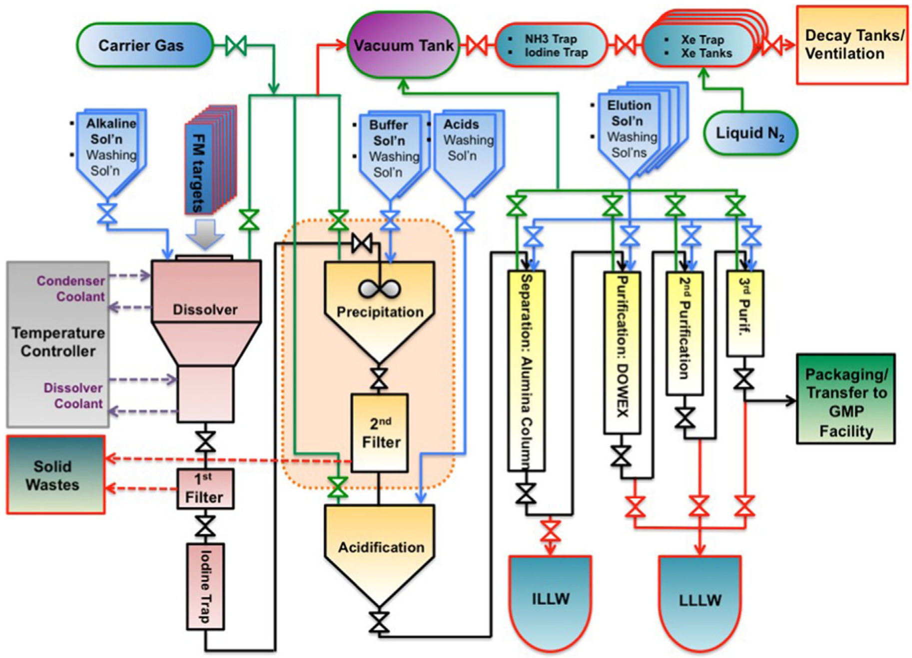

Fig. 1. Process scheme of KJRR fission ^{99}Mo production and types of radioactive waste [8].

Among the various methods of solidifying radioactive waste are cementation, bituminization, and vitrification. Cementation is the oldest method in the solidification of radioactive waste, and its technical stability and convenience have already been verified [11]. Secondary waste is not produced since the process does not involve heat. The simple, compact process requires a relatively small installation space, and is easy to operate and maintain [12,13]. A disadvantage of the cementation process is that it produces more solids than other approaches. In the case of ILLW, intermediate-level nuclides are dispersed in cement waste form, allowing them to be economically disposed in low-level facilities while ensuring radiation protection. In a previous study, our team showed that cement waste form samples derived from mixing alumina cake and ILLW, which were generated by KJRR's FMPP, satisfy the WAC of KORAD [14].

Operators of radioactive waste treatment facilities (RWTF) are expected to establish processing methods and operating conditions for different types of solid and liquid wastes generated from FMPP. In addition, the processed radioactive wastes should satisfy the WAC of disposal facilities.

The purpose of this study is to first determine the operating range and optimum operating conditions for the cementation of ILLW from FMPP. Second, it seeks to evaluate the disposal feasibility of cement waste form produced under optimum operational conditions. For this purpose, the operating range of cementation was set with reference to the water-to-cement (w/c) ratio, which should be suitable for the preparation of cement waste form and not produce free standing water. Optimum operational conditions corresponding to the operating range were derived in consideration of economic feasibility. This study evaluated the structural stability of cement waste form prepared under optimum operational conditions, as well as the leaching stability of nuclides. As specified in KORAD's WAC, the evaluation was comprised of a compressive strength test, thermal cycling test, irradiation test, water immersion test, and leaching test.

2. Materials and methods

2.1. Materials

2.1.1. Simulated liquid waste of ILLW

The simulated liquid waste of ILLW used in the experiment was the waste liquid produced from the inactive pilot-plant experiment of FMPP. The major components of ILLW are presented in Table 1 [15]. The amount of ILLW produced from KJRR is expected to be 3000 L in a year, and the concentration of Na_2SO_4 and NaCl is 106.53 g/L and 64.28 g/L, respectively. At 20 °C, the solubility of Na_2SO_4 and NaCl is 139 g/L and 359 g/L, meaning that they exist in the dissolved state in ILLW. Na^+ is produced due to NaOH, used in the alkaline dissolution of uranium metal. SO_4^{2-} and Cl^- originate from H_2SO_4 and HCl, which are used to acidify solutions for the purification of Mo-99.

Since ILLW produced by FMPP has high radioactivity, it is first stored for five years to allow natural attenuation of short-lived nuclides. Cs-137 ($t_{1/2}$ = 30.17 yr), which has a radioactive concentration of 6.53 × 106 Bq/mL and releases gamma rays, is classified as ILLW despite its relatively long half-life. Table S1 shows the radioactive nuclides of ILLW and their concentrations after five years [16].

2.1.2. Cement

Cement is classified into type I to V depending on the weight ratios of 3CaO SiO_2, 2CaO·SiO_2, 3CaO·Al_2O_3, and 4CaO·Al_2O_3·Fe_2O_3. Table S2 shows the classification of cement based on Korean Industrial Standards (KS) [17]. Depending on type, cement has different characteristics, including heat of hydration, hardening rate, and resistance to sulphate. Type I Portland cement, which is the most widespread, was purchased from SsangYong C&E (South Korea), and used in the preparation of cement waste form samples.

Table 1
Chemical characteristics of KJRR ILLW [13].

Chemical composition	Concentration (g/L)	Solubility in water at 20 °C (g/L)	Generation rate (L/yr)
Na_2SO_4	106.53	139	3000
NaCl	64.28	359	

2.2. Preparation of cement waste form

Cement and LLW (simulated liquid waste) were mixed in accordance with "Testing method for mechanical mixing of hydraulic cement pastes and mortars of plastic consistency" (KS L 5109) [18] using a mechanical mixer (JI-206, JEIL, Korea). The mixtures were poured into polyethylene molds (D = 5 cm, H = 12 cm), which were covered with lids to prevent moisture from evaporating. They were then cured for 28 days at room temperature (18–25 °C) and a relative humidity range of 30–60%. The amount of free standing water remaining in the molds after curing was measured using a graduated pipette. Next, the molds were carefully removed to prevent damage to the samples, which were fabricated to have a diameter of 50 mm and height of 100 mm.

The prerequisites of preparing cement waste form samples are the mixability of cement and ILLW (simulated liquid waste) and flowability of mixtures. The mixability generally depends on the w/c ratio. Therefore, floability is determined by the w/c ratio of liquid waste using the "Flow table for use in tests of hydraulic cement" (KS-L-5111) [23]. The w/c ratio was varied from 3.79 to 6.57 to examine its effect on the flowability of mixtures comprised of ILLW (simulated liquid waste) and cement.

2.3. Evaluation of disposal feasibility of cement waste form

The disposal feasibility of cement waste form samples prepared using mixtures of cement and ILLW (simulated liquid waste) was evaluated based on the test items and methods specified in the WAC of KORAD. Table 2 presents the test items and methods in KORAD's WAC [19]. Detailed test procedures can be checked through the standard methods in Table 2 [20,24–28]. Here, structural stability is evaluated after 28 days of curing through an initial compressive strength test, water immersion test, thermal cycling test, irradiation test, and compressive strength test (after water immersion, thermal cycling, and irradiation tests), and the criterion to be met is 35.2 kgf/cm^2 (3.44 MPa). Three cement waste form samples were prepared for each test item, and the average values for the three samples were used in the evaluation.

Introduced to ILLW (simulated liquid waste) such that the concentration of Cs, Co, and Sr was 2000 ppm each. Three leaching samples were prepared, and the average values for the three samples were used in the evaluation. The leachability of cement waste form was evaluated based on the leachability Index (*LX*). Here, *LX* is defined as follows.

$$LX = -\log D_e \tag{1}$$

D_e = effective diffusion coefficient [cm^2/sec]

$$f(t) = CFL = \frac{\sum a_n}{A_0} = 2\frac{S}{V}\left[\frac{D_e}{\pi}\right]^{1/2} \$ t^{1/2} \tag{2}$$

Based on Eq. (2), the plot of $\left[\frac{\sum a_n}{A_0}\right]$ with respect to $[\sqrt{t}]$ has the following slope:

$$\text{Slope} = 2\frac{S}{V}\left[\frac{D_e}{\pi}\right]^{1/2}$$

By substituting slope into Eq. (3), D_e can be calculated as follows.

$$D_e = (slope)^2 \$ \left[\frac{1}{2}\frac{V}{S}\right] \$ \pi \tag{3}$$

CFL = cumulative fraction leached; a_n = the total amount of material released during leaching periods up to time *t*; A_o = the initial amount of material; *V* = waste form volume; *S* = the surface area of waste form.

3. Results and discussion

3.1. Operating range and optimum operational conditions

The amount of cement waste form requiring disposal decreases with increasing ILLW content. The ratios of cement and ILLW determine not only the workability of the cementation process, but also the characteristics of cement waste form. A low ILLW content hinders mixing, whereas excess ILLW results in free standing water, making samples inappropriate for disposal. Thus, cement waste form samples were prepared with varying w/c ratios to determine the optimum mixing ratio and operating range.

Fig. 2 shows the cement waste form samples containing ILLW (simulated liquid waste) and prepared with varying w/c ratios, and Fig. 3 presents the mixability limit, free standing water limit, and operating range of ILLW (simulated liquid waste) and cement in relation to w/c ratio. The mixing of ILLW and cement was possible at w/c ratios of 0.30 and 0.35, but the lack of flowability left traces of bubbles on the surface of samples. Sufficient mixability and flowability for producing a cement waste form were achieved from a w/c ratio of 0.4 onwards, continuing in the range of 0.40–0.70. As the

Table 2
Test items and methods specified in KORAD's WAC [17].

Item	Test	Standard method	Test method	Criteria
Structural Stability	Compressive strength test	KS F2405 [24]	–	≥35.2 kgf/cm^2 (3.44 MPa)
	Water immersion test (90 days)	NRC[a] [20]	Compressive strength after immersion test	≥35.2 kgf/cm^2
	Thermal cycling test (28 days)	ASTM B553 [25]	Compressive strength after thermal cycling test	≥35.2 kgf/cm^2
	Irradiation test	NRC[a] [20]	Compressive strength after irradiation (1.0x10^{7}Gy)	≥35.2 kgf/cm^2
Leachability	Leaching test (90 days)	ANS 16.1 [26]	Cs, Sr, Co	Leachability Index ≥6
Free standing water test	Sample	EPA[b] [27]	–	>0.5 vol%
	200 L drum	ANS 55.1 [28]	–	>0.5 vol%

[a] NRC, Waste Form Technical Position, Revision 1. (1991).
[b] EPA, Method 9095B "Paint Filter Liquids Test"

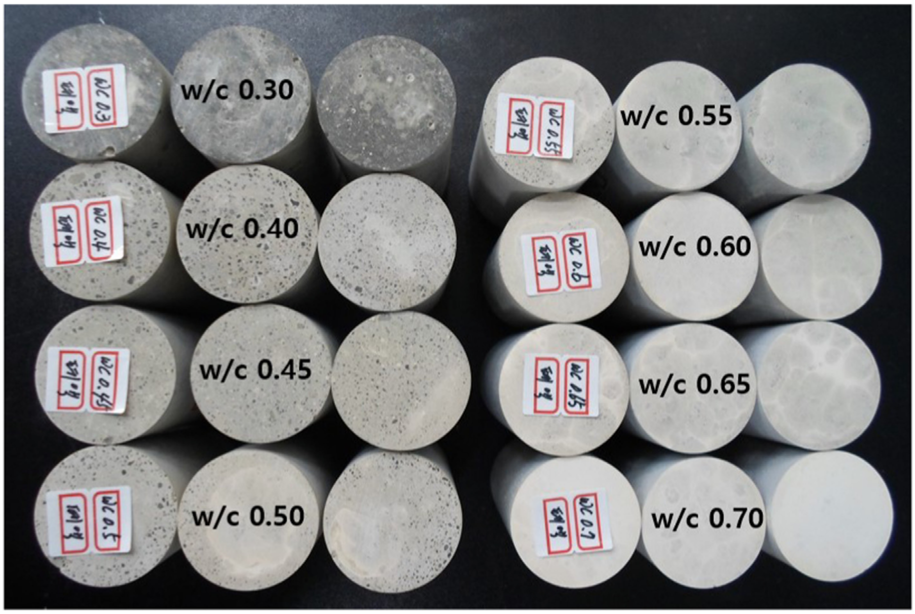

Fig. 2. Cement waste form samples prepared using ILLW (simulated liquid waste) and Portland cement (type I).

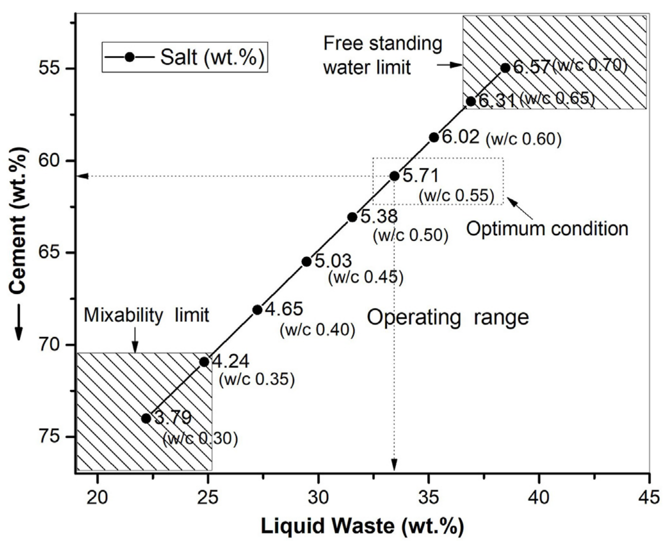

Fig. 3. Operating range and optimum operational conditions of cement waste form.

content of liquid waste corresponding to w of w/c ratio increases, the waste volume can be reduced. However, if the w of w/c ratio is too high, free-standing water may occur after producing cement waste form. Therefore, the optimum w/c range was determined considering the condition in which free-standing water does not occur. When the w/c ratio was 0.65 and 0.7, the cured samples contained free standing water. As such, the optimum operating range for the preparation of cement waste form samples was at a w/c ratio from 0.40 to 0.60.

The amount of cement waste form decreases when cement mixtures contain more ILLW. That is, the most economically feasible w/c ratio is 0.60. However, considering variables such as precision of cementation equipment and skill level of workers, optimum operational conditions were achieved at a w/c ratio of 0.55.

3.2. Evaluation of disposal feasibility of cement waste form

The disposal feasibility of cement waste form was evaluated with cement waste form samples prepared at the optimum w/c ratio of 0.55. The evaluation, conducted after 28 days of curing, was comprised of an initial compressive strength test, thermal cycling test, irradiation test, water immersion test, compressive strength test, and leaching test.

Fig. 4 shows the changes in volume and weight of the samples before and after the thermal cycling test, irradiation test, water immersion test, and leaching test. The weight of samples decreased by 12.40%, 12.73%, 6.59%, and 4.56%, respectively, after the thermal cycling test, irradiation test, water immersion test, and leaching test. The decrease in weight can be attributed to the characteristics of each test, resulting from the loss of moisture within samples and the elution of ions. The change in the volume fraction of the cement

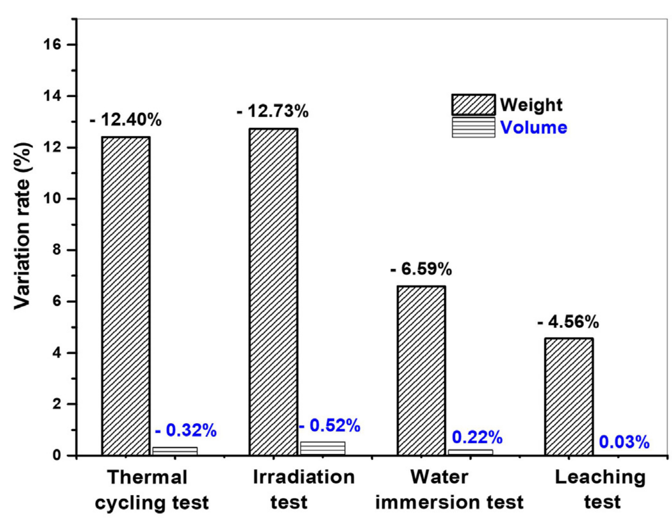

Fig. 4. Weight and volume change of cement waste form.

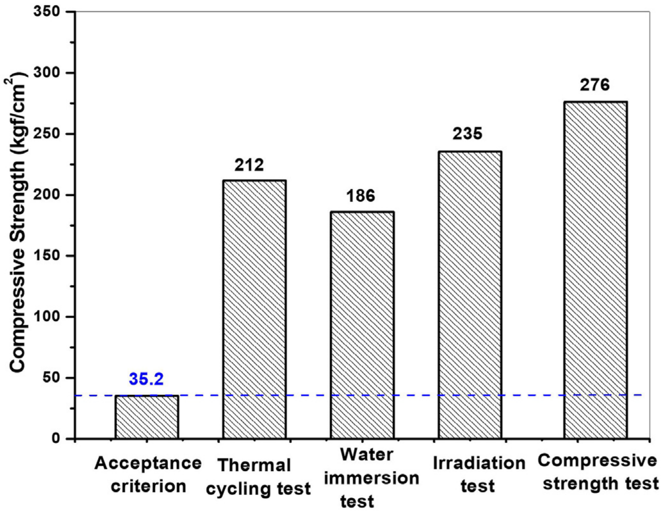

Fig. 5. Compressive strength of cement waste form samples before and after the thermal cycling test, water immersion test, and irradiation test.

waste form was calculated by checking the density and weight change of the cement waste form. The volume of samples decreased by 0.32% and 0.52% after the thermal cycling test and irradiation test, and increased by 0.22% and 0.03% after the water immersion test and leaching test. These changes in volume can be seen as falling in the range of experimental error, and thus having no effect on the structural stability of samples. This presumption is consistent with the results of compressive strength measurements taken after each test.

Fig. 5 shows the compressive strength of cured samples before and after the thermal cycling test, water immersion test, and irradiation test. Fig. 6 presents the actual photos of samples during the compressive strength test. The initial compressive strength of cement waste form samples after curing was 276 kgf/cm^2 (27.06 MPa), about eight times larger than the acceptance criterion of 35.2 kgf/cm^2 (3.45 MPa). The compressive strength was 212 (20.79), 186 (18.24), and 235 (23.04) kgf/cm^2 (MPa) after the thermal cycling test, water immersion test, and irradiation test, thus satisfying the acceptance criterion of the disposal facility.

The Nuclear Regulatory Commission (NRC) recommends testing the stability of cement waste forms through 180 days water immersion test if the compressive strength of cured samples is less than 75% of initial values following 90 days water immersion test [20]. The compressive strength of samples after 90 days water immersion test was 186 kgf/cm^2, less than 75% of the initial compressive strength of 276 kgf/cm^2. As such, 180 days leaching test was performed. Fig. 7 shows the compressive strength after 90 and 180 days of immersion. The samples were free of cracks or collapses after 180 days, and their compressive strength was maintained.

Fig. 8 shows the cumulative leached fraction of Cs, Sr, and Co in relation to time based on the leaching test. The plots show an almost linear distribution, indicating that the leaching of Cs, Sr, and Co in cement waste form follows the typical diffusion model [14]. The slope of Cs was much steeper than that of Sr and Co. This means that Cs has a faster leachability than the other two nuclides [21,22]. The slopes representing D_e were substituted into Eq. (1) to calculate the *LX* in Fig. 9.

The *LX* of Cs, Sr, and Co in cement waste form must be above 6 (Table 2) to satisfy the WAC of KORAD. The average *LX* of Cs, Sr, and Co was 8.36, 10.41, and 12.30, thus verifying the high leaching resistance and disposal feasibility of cement waste form. The *LX* of Na, a major component of ILLW but not specified in WAC, was 8.48.

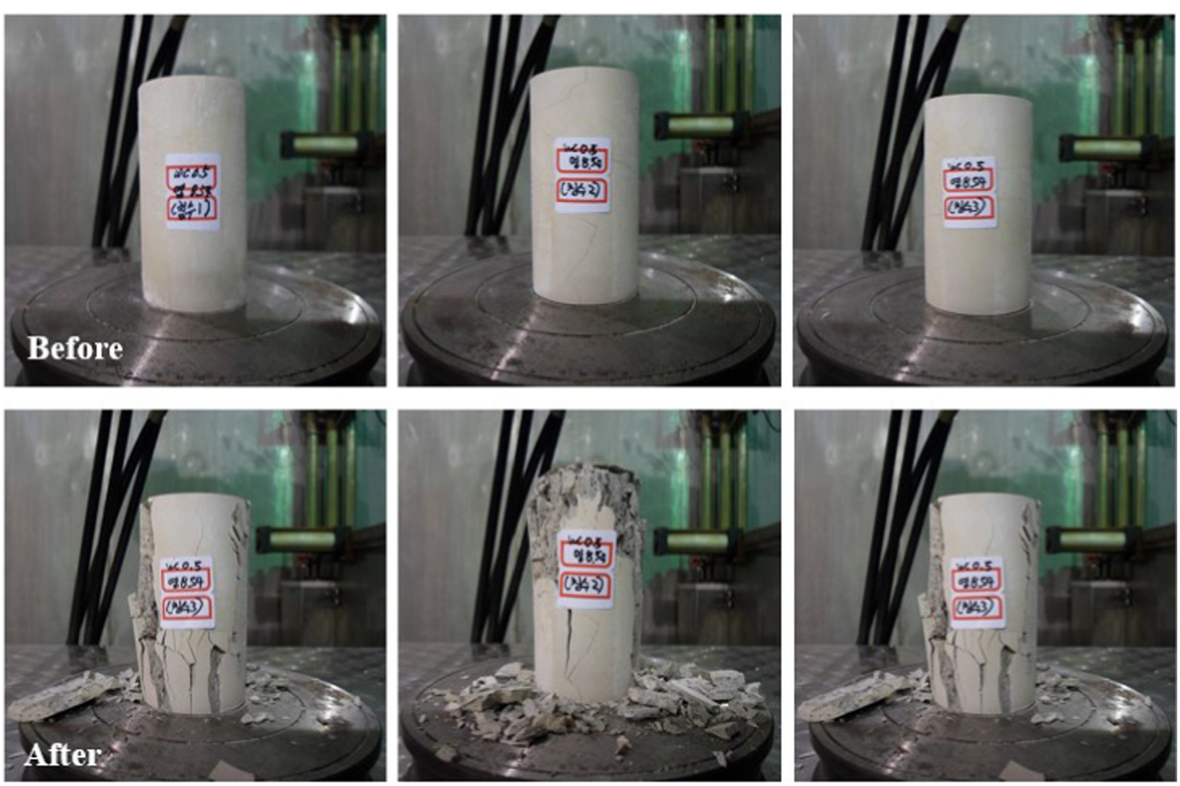

Fig. 6. Appearance of samples in the compressive strength test after water immersion.

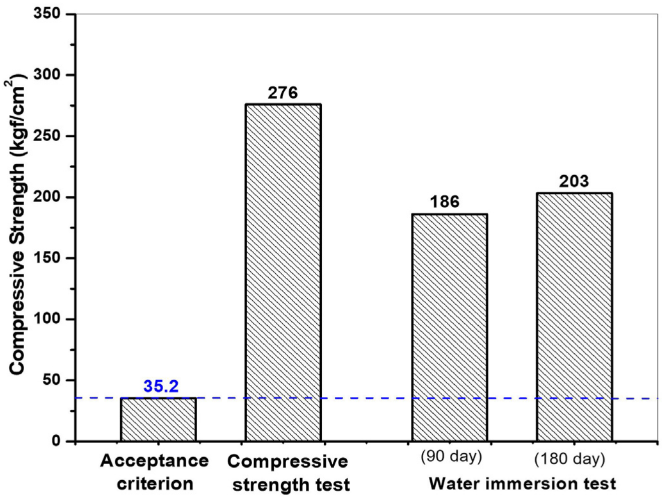

Fig. 7. Compressive strength after 90 days and 180 days of water immersion.

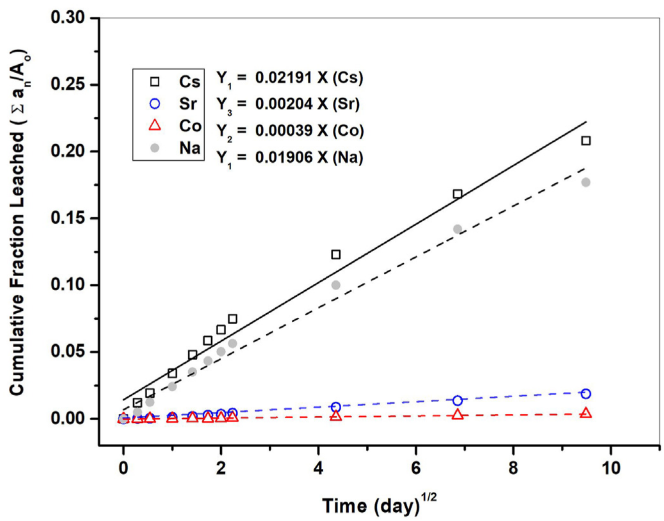

Fig. 8. Cumulative fraction of Cs, Sr, Co leached from cement waste form.

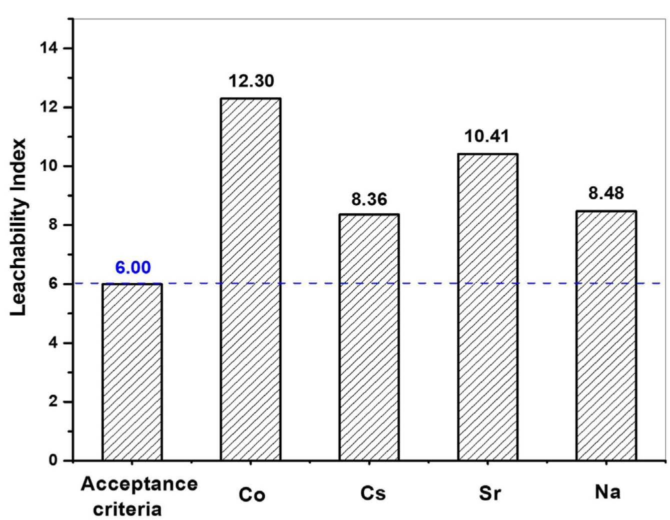

Fig. 9. Leachability index (*LX*) of Cs, Sr, and Co nuclides in cement waste form samples.

4. Conclusions

This study determined optimum conditions for the cementation of ILLW from FMPP, and evaluated the disposal feasibility of cement waste form samples. The range of w/c ratios for ILLW cementation was 0.4–0.6. Considering the economic feasibility and stability of cementation, optimum operating condition were achieved at a w/c ratio of 0.55. Here, the corresponding salt content was 5.71 wt%. The cement waste form samples prepared under optimum operational conditions had initial compressive strength of 276 kgf/cm^2. After the thermal cycling, the water immersion, and the irradiation tests, compressive strength of the samples was confirmed to be 212, 186, and 235 kgf/cm^2, respectively. These values exceeded the criterion of 35.2 kgf/cm^2 specified in KORAD's WAC. In addition, the leachability index (*LX*) of Cs, Sr, and Co nuclides was 8.36, 10.41, and 12.30. These values were higher than the acceptance criterion of 6, thus verifying the disposal feasibility of the samples. The results indicate that the proposed cementation conditions for the disposal of ILLW from FMPP can be effectively applied to KJRR's disposal facility.

CRediT author statement

Jong-Sik Shon: conceptualization, investigation, original draft preparation. **Hyun-Kyu Lee**: investigation, writing-review and editing. **Tack-Jin Kim**: visualization, writing-reviewing. **Gi-Yong Kim**: resources. **Hongrae Jeon**: methodology, investigation.

Declaration of competing interest

The authors declare that they have no known competing financial interests or personal relationships that could have appeared to influence the work reported in this paper.

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Appendix A. Supplementary data

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