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PROPERTIES OF CEMENT-SOLIDIFIED SOILS FOR THE PROCESS CONTROL PROGRAM

The performance of cement-solidified soil waste was evaluated following Process Control Program (PCP) guidelines. Illite-based soils were used as surrogate for radioactive soil waste. The optimal mixing ratio of water, cement, and waste was determined from compression strength and workability; the solidified block at a mixing ratio of 25 wt.% water, 50 wt.% soil, and 25 wt.% cement exhibited 12.7 MPa of compression strength with good workability. Cement-solidified soil blocks in PCP size (diameter: 100 mm; height: 200 mm) were fabricated at the determined mixing ratio. Compression, immersion, thermal cycling, and irradiation tests were performed on the solidified blocks. The cement-solidified blocks passed the acceptance KORAD criteria for disposal, satisfying a compression strength above 3.44 MPa after all tests. Leaching tests were performed by piking radioactive natural soils (39.36 Bq/g), showing no radioactivity. These results suggest that radioactive soil waste can be solidified with cement by following PCP guidelines.

Keywords: Solidification; soil; cement; Process Control Program; disposal

1. Introduction

For radioactive waste, nuclide immobility must be ensured via encapsulation and fixation using suitable solidification media that are deliverable to low- and intermediate-level radioactive waste disposal facilities. The Korea Radioactive Waste Agency (KORAD) has released acceptance criteria for underground repositories of low- and intermediate-level radioactive waste [1], which recommend that homogeneous waste, such as spent resins and sludges, be solidified. Furthermore, the cement solidification Process Control Program (PCP) was disclosed as a guideline [2]. Guidelines recommend confirming the chemical and physical performance of solidified waste.

The PCP contains process and test methods for the quality assurance of solidified products to guarantee delivery decisions in the KORAD depository. Fig. 1 shows the PCP establishing process for each target waste, consisting of two main steps. The first step is waste and solidification matrix characterization. Waste characteristics include radioactivity and waste steam history. The solidification matrix characteristics are fundamental properties such as density, composition, crystal structure, chemical properties, and solidification mechanism. The next step is process variable development for solidification performance evaluation. Evaluation methods are established based on the KORAD waste acceptance criteria. The detailed method for each measurement can be found in the literature [1,3-6]; the compression strength should be above 3.44 MPa after curing, immersion, thermal cycling, and radiation tests, and the solidified products should pass reaching testing.

The Korea Atomic Energy Research Institute (KAERI) stores uranium-contaminated soil produced during fuel conversion facility decommissioning. Soil radioactivity was characterized as ~6.30×10^2 Bq/g, and the activity was categorized as low-level radioactive waste according to the Korean regulations [7]. Although soils are chemically very stable under depository conditions, fine soils below 0.2 mm should be solidified to ensure nuclide immobility from the wastes. Cement is a common solidification matrix widely used as a structural material, and is applicable to hazardous waste solidification [8,9]. Therefore, studying the properties of cement-solidified soils for radioactive waste disposal is important.

The main objective of this study was to evaluate the performance of cement-solidified soil waste. In this study, all tests were performed following PCP guidelines. Illite-based soils

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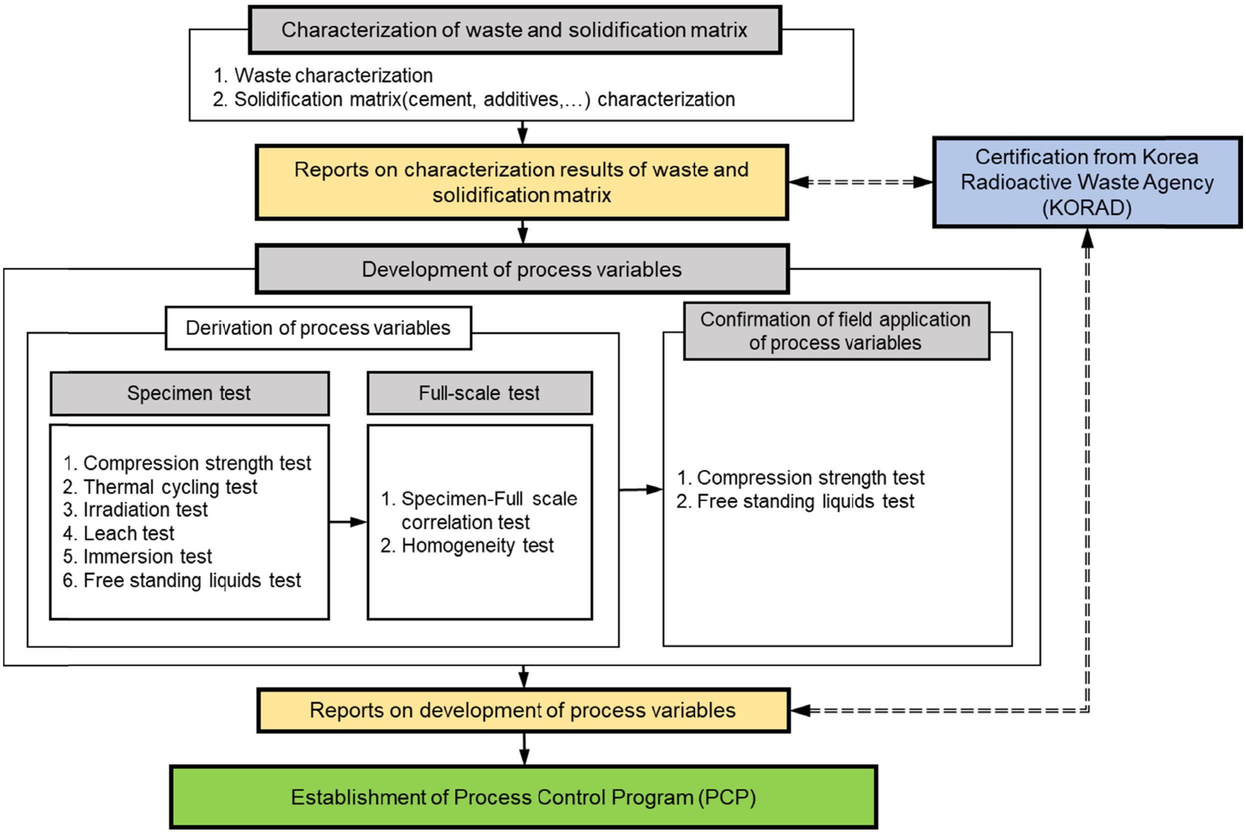

Fig. 1. Process for cement solidification Process Control Program (PCP) establishment in the Republic of Korea

were used as a surrogate for radioactive soil waste. The optimal mixing ratio of water, cement, and waste was determined from the compression strength and workability of samples with a size of 24 mm prepared by changing the mixing ratio, and the compression strength was measured after 7 d of curing. Following PCP guidelines, 100-mm cement-solidified soil blocks were fabricated at the determined mixing ratio. Compression, immersion, thermal cyclic, irradiation, and leaching tests were performed on the solidified blocks to evaluate their physical stability. Leaching tests were performed by piking radioactive natural soils (39.36 Bq/g) from Goesan, Republic of Korea, and chemical stability was confirmed. Repository acceptance was confirmed based on these results.

2. Materials and Methods

2.1. Solidification

Illite-based soils (Yongkoong Illite Co., Ltd., after sieving with 18 mesh) were purchased as a surrogate for uranium-contaminated soils, based on preliminary tests to determine a surrogate soil. The X-ray diffraction (D2 Phaser, Bruker) patterns for both the actual uranium-contaminated soils and illite-based soil samples exhibited similar trends, and the X-ray fluorescence (Delta professional, Olympus) analysis supported these results (not shown here). Portland cement (KS L 5201, Ssang Yong C&E Co., Ltd.) was used as the solidification matrix.

In Lab-scale solidification, the soil, cement, and distilled water were weighed prior to mixing. The cement was poured into the water in a jar; then, soil was added. The mixture was stirred with a rod and poured into a 24-mm-diameter plastic mold with a cap. The diameter-to-height ratio of the samples was maintained at 2. Four samples were produced by mixing to assess reproducibility. After 7 d of curing, the cement-solidified soils were retrieved and used for compression strength measurements. In PCP-scale solidification, the cement slurries were mixed using a rotary mixer (KDPI-720, KD Precision Co., Ltd.). Water (925 g), soil (1850 g), and cement (925 g) were weighed before mixing. They were then poured into the mixer container and operated for approximately 3 min. The slurry was poured into a 100-mm-diameter, 200-mm-high plastic mold (HJ-3190, Heungjin). The top was sealed with Parafilm during curing. After 28 d of curing, the samples were used for performance testing.

2.2. Performance tests

A compression testing machine (QREC-81, QURO) calibrated and certified by the Korean Industrial Standards (KS), was used for the compression strength test, which was performed following the KS F2405 standard procedure [3]. Immersion tests were performed following standard procedure [4]. The block was immersed into 9 L of distilled water for 90 days and dried for 2 days at room temperature. Thermal cycle tests were performed using a temperature-programmed oven (WTC-288, WON Technology Co.) following ASTM B533 [6]. The oven was heated to 60°C at a ramping rate of 10°C/h, and the temperature was maintained for 1 h. The oven was then cooled to –40°C at a cooling rate of 10°C/h, and the temperature was maintained for 1 h. The heating and cooling processes were repeated 30 times. Irradiation testing was carried out with a gamma-ray irradiator (Jeongeup, KAERI) upto 10^7 Gy of irradiation at a dose rate of 1.2×10^4 Gy/h. Morphological changes and compression strength were observed after all the tests. Leaching tests were conducted using ANS 16.1 [5]. These tests were conducted by piking radioactive natural soils (39.36 Bq/g) sampled from Goesan. Approximately 150 g (~5870 Bq) of Goesan soil was added. The solidified cement blocks were inserted into 7.9 L of distilled water. The water-to-surface-area ratio was approximately 10.15 cm, following test requirements (≤10.2 cm). The leachate was collected after rinsing for 2 h, 7h, 1, 2, 3, and 5 days. They were characterized using HP-Ge (GC3018, Canberra Industry).

3. Results and discussion

The optimal mixing ratio of water, cement, and soil waste was determined via lab-scale solidification tests, to simultaneously maximize the soil loadings and obtain a compression strength above 3.44 MPa. After determining the mixing ratio, the cement-solidified soil properties were evaluated according to PCP guidelines.

3.1. Optimum soil mixing ratio in cement solidification

The workability and compression strength of the cured block samples were measured for various mixing ratios. The green circles in the ternary graph in Fig. 2 correspond to the test conditions for the optimum water, cement, and soil waste ratio. The water, soil, and cement amounts varied in the ranges of 20-30, 15-80, and 0-60 wt.%, respectively. The test conditions marked with a red cross correspond to those in which the cement-soil slurries did not mix, as shown in the inset figure, indicating that the conditions are not applicable for solidification. The well-mixed slurries were solidified into lab-scale block samples and used for the compression strength test, as shown in Fig. 3(a). The compression strength decreased as the soil and water content increased. It should be noted that the maximum soil loading was 50 wt.%, confirming a strength above 3.44 MPa (the acceptance

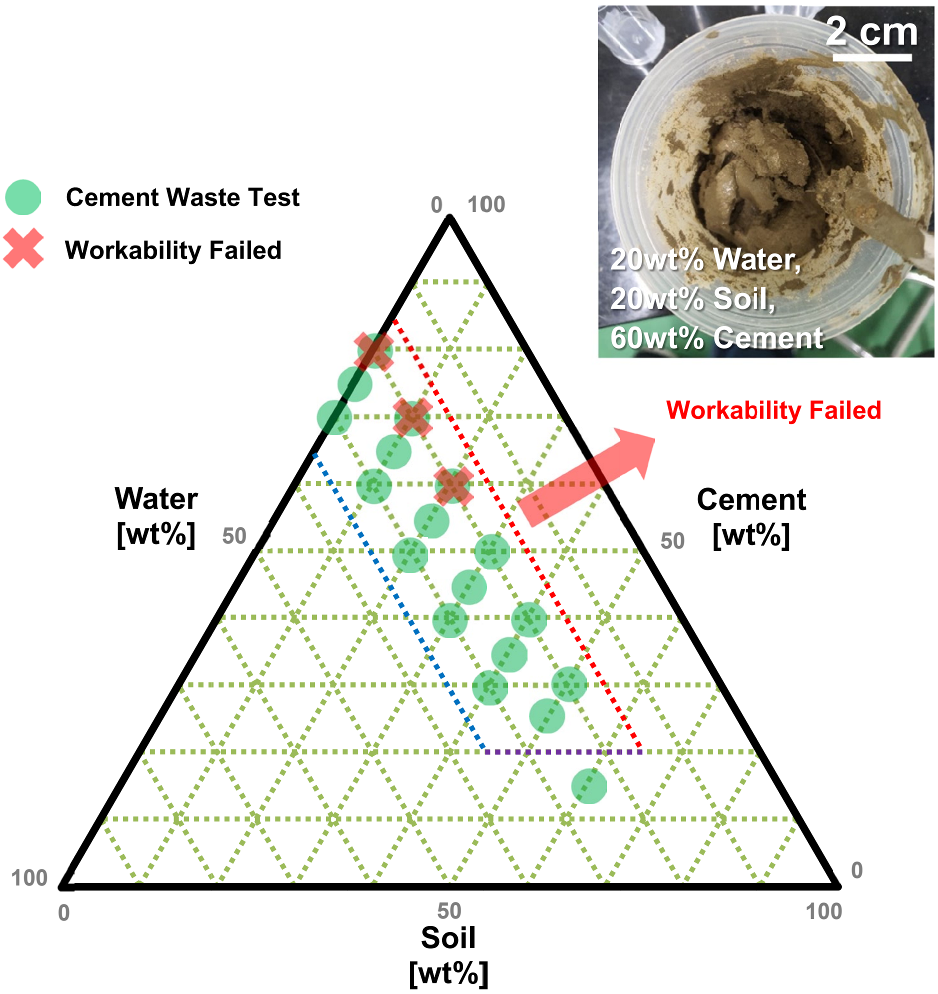

Fig. 2. Test conditions for the cement-solidification of soils at various cement, water, and soil mixing ratios. The green circles and red crosses correspond to the tested and workability failed conditions, respectively

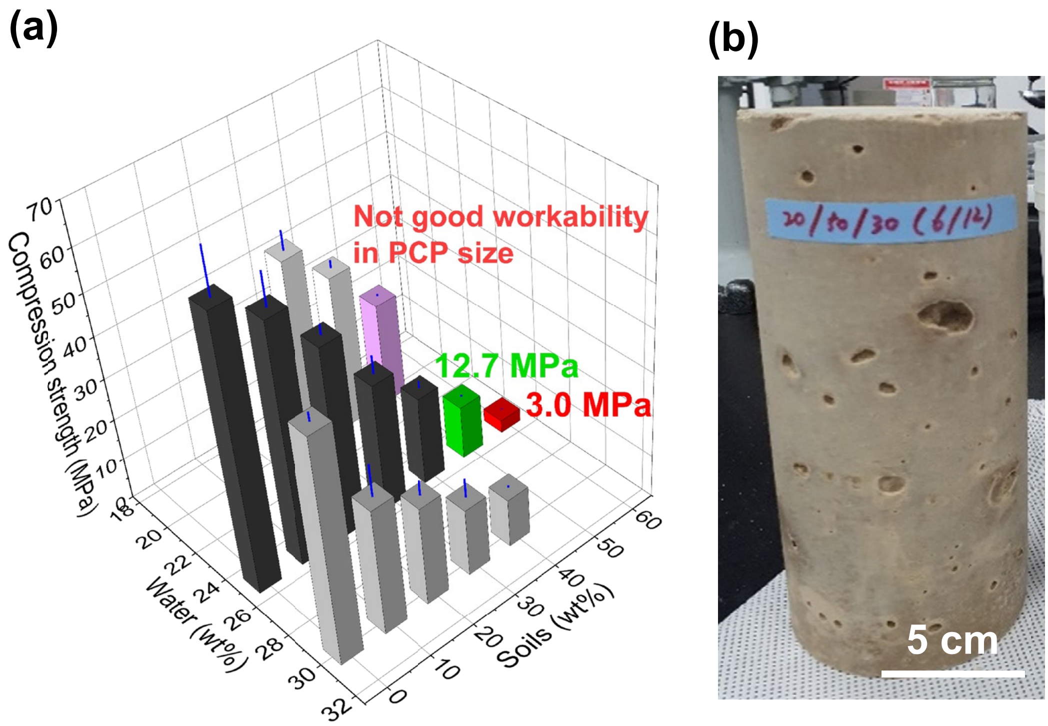

Fig. 3. (a) Compression strength of cement-solidified soils at various cement, water, and soil mixing ratios, and (b) a photograph of a sample in PCP-scale at a mixing ratio of 20-wt.% water, 50-wt.% soil, and 30-wt.% cement

criteria). Under the 25-wt.% water and 60-wt.% soils condition, the compression strength was just below the acceptance criteria, showing 3.0 MPa of strength; under the 20-wt.% water and 50-wt.% soils condition, even though it exhibited high compression strength (purple bar in Fig. 3(a)), the workability was insufficient for PCP-scale sample preparation, exhibiting pores after solidification (see Fig. 3(b)). Thus, the optimum soil mixing ratio for cement solidification was determined to be 25 wt.% water, 50 wt.% soils, and 25 wt.% cement.

3.2. Properties of cement-solidified soils following PCP guidelines

The compression strength of the solidified soils was measured after curing, immersion, thermal cyclic, and irradiation tests on the PCP scale to ensure physical stability, as shown in Fig. 4(a). After 28 d of curing, the strength was measured three times to ensure reproducibility. The results were compared with those obtained at a laboratory scale. The PCP-scale solidified soils exhibited lower strength compared to that of the lab-scale

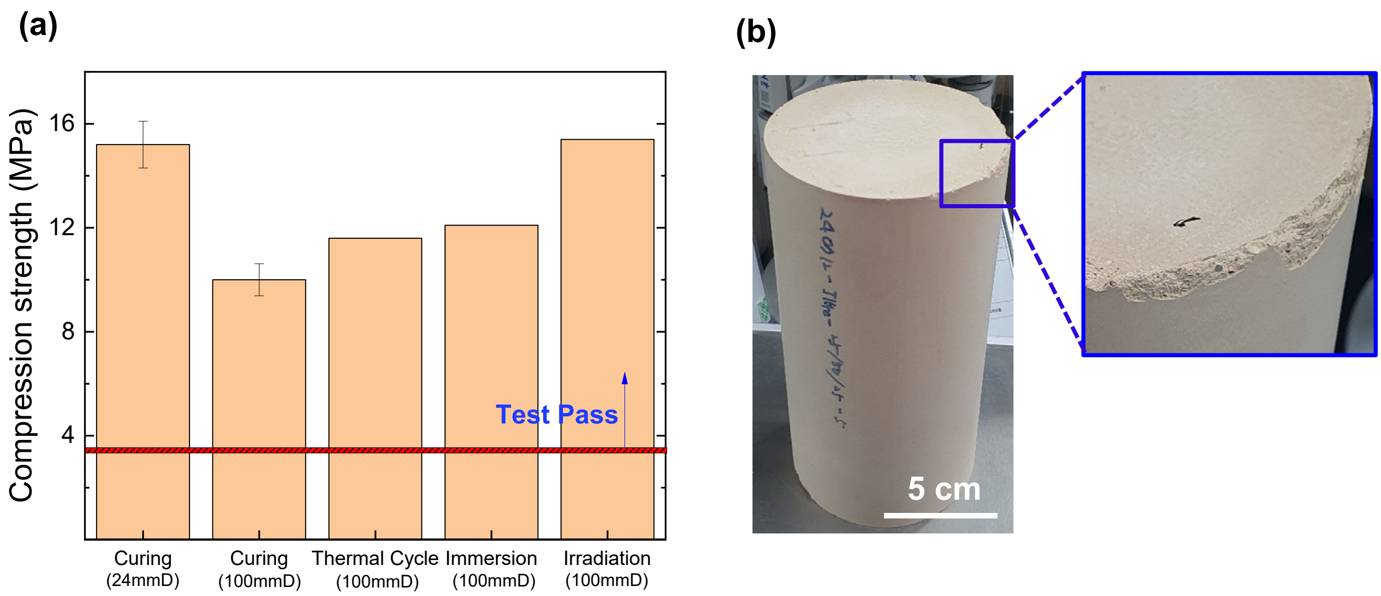

Fig. 4. (a) Compression strength of PCP-scale cement-solidified soils after curing, thermal cycling, immersion, and irradiation tests, and (b) a photograph of a sample after the irradiation test

soils, decreasing 15.2 to 10.0 MPa via scale-up. It is considered that the heat from the cement hydration in the PCP scale provokes localized fast solidification, which is attributed to the weak structural properties [10,11]. After 90 d of immersion, no significant change in appearance was observed, and the compression strength increased to ~12 MPa after testing. We believe that both the additional curing time and hydration of the remaining cement during immersion caused the strength increase. The thermal cycling test results are shown in Fig. 4(a). No significant change in appearance is observed after testing, and the compression strength increased to ~12 MPa, similar to the immersion test results. After irradiation testing, the solidified block successfully maintained a clean surface; however, only a small part of the edge in the block was peeled off as shown in Fig. 4(b). The compression strength increased to ~15 MPa, revealing that the irradiation did not degrade the cement-solidified soils but increased the strength.

To confirm the chemical stability of the solidified block, leaching tests were performed after spiking natural uranium-containing soil. The leachate radioactivity was measured; however, no activity was detected under any condition. The excellent chemical stability suggests that cement solidification even with actual radioactive soil wastes will maintain the stable performance, exhibiting a high leachability index (above 6). This is reasonable because natural uranium exists mainly as a mineral in ores [12]. Practically, the target nuclides for leaching testing in the PCP guidelines are Cs, Sr, and Co, which are known as mobile elements because of their solubility in water; however, no guidelines have been suggested for the leaching test of uranium.

These results confirm that the solidified cement soils satisfy all PCP requirements: the compression strength was above 3.44 MPa in all tests (compression, immersion, thermal cycling, and irradiation tests), and no activity was detected in the leaching test.

4. Conclusions

In this study, the performance of cement-solidified soils was evaluated following PCP guidelines. Based on the results, the main conclusions of this study can be summarized as follows:

- The optimal mixing ratio was determined to be 25 wt.% water, 50 wt.% soil, and 25 wt% cement, exhibiting 12.7 MPa of compression strength with good workability.
- Compression strength of the cement-solidified soils was above 3.44 MPa in the compression (~10 MPa), immersion (~12 MPa), thermal cycling (~12 MPa), and irradiation tests (~15 MPa).
- No radioactivity was detected after the leaching test of spiking natural uranium-contained soils.

These results confirm that the cement-solidified soils satisfy all PCP requirements. In future work, a full-scale test must be conducted to evaluate the feasibility of cement solidification in a practical 200-ℓ drum.

Acknowledgments

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