Nuclear Engineering and Technology 55 (2023) 876–883

Contents lists available at ScienceDirect

Nuclear Engineering and Technology

journal homepage: www.elsevier.com/locate/net

Original Article

Statistical analysis of effects of test conditions on compressive strength of cement solidified radioactive waste

Hyeongjin Byeon, Jaeyeong Park*

Department of Nuclear Engineering, Ulsan National Institute of Science and Technology, 50 UNIST-gil, Ulsan, 44919, Republic of Korea

ARTICLE INFO

Article history:
Received 3 July 2022
Received in revised form
17 October 2022
Accepted 16 November 2022
Available online 19 November 2022

Keywords:
Cement solidified specimen
Compressive strength
Loading rate
Radioactive waste
Statistical analysis

ABSTRACT

Radioactive waste should be solidified before being disposed of in the repository to eliminate liquidity or dispersibility. Cement is a widely used solidifying media for radioactive waste, and cement solidified waste should satisfy the minimum compressive strength of the waste acceptance criteria of a radioactive repository. Although the compressive strength of waste should be measured by the test method provided by the waste acceptance criteria, the method differs depending on the operating repository of different countries. Considering the measured compressive strength changes depending on test conditions, the effect of test conditions should be analyzed to avoid overestimation or underestimation of the compressive strength during disposal. We selected test conditions such as the height-to-diameter ratio, loading rate, and porosity as the main factors affecting the compressive strength of cement solidified radioactive waste. Owing to the large variance in measured compressive strength, the effects of the test conditions were analyzed via statistical analyses using parametric and nonparametric methods. The results showed that the test condition of the lower loading rate, with a height-to-diameter ratio of two, reflected the actual cement content well, while the porosity showed no correlation. The compressive strength assessment method that reflects the large variance of strengths was suggested.

1. Introduction

Radioactive waste with liquidity or dispersibility, which can be easily diffused in the repository, should be solidified using materials like cement or polymer before being transferred to the repository. Solidified waste disposed of in the repository should maintain its structural stability under predicted and disposal conditions. Therefore, the criteria for solidified waste differ for different countries based on their operating disposal facility, owing to different repository types or disposal conditions. The features of solidified waste and the conditions that solidified waste should endure are clarified in the waste acceptance criteria (WAC) of low- and intermediate level waste (LILW) or low-level waste (LLW) repository.

The compressive strength of solidified waste is a major factor in WAC that prevents the physical deformation of waste [1]. A large enough compressive strength can prevent damage in the long term, and its stability can delay the leaching of radionuclides from solidified waste to the biosphere by preventing the increase of the surface area of the waste form [2,3]. Therefore, Korea and U.S. require a compressive strength of 3.44 MPa for cement solidified waste in repositories, which should satisfy the same compressive strength under expected disposal conditions (e.g., irradiation, immersion, and thermal cycling) [4,5]. However, factors such as the type, dimension, and test method of specimens can significantly affect the compressive strength of solidified waste [6–10].

Factors that affect the compressive strength of concrete specimen are height-to-diameter (H/D) ratio, loading rate, and porosity. The compressive strength of concrete specimens differs depending on the H/D ratio [11,12]. The measured compressive strength of a specimen is higher when the loading rate is higher, which causes a ~30% difference. The compressive strength of concrete specimen differs according to the porosity; that is, higher porosity can decrease the compressive strength [7,13]. In "NRC, technical position on waste form, rev 1," the compressive strength of solidified waste should be tested by the ASTM C39 which is for cylindrical shape specimen. In ASTM C39, the weighting factor should be applied when the H/D ratio is not two [14]. As mentioned above, although several factors affect the compressive strength of solidified waste, the effect of these factors on specimen having the same amount of sludge or solidifying media has not been evaluated for

* Corresponding author.
E-mail address: jypark@unist.ac.kr (J. Park).

https://doi.org/10.1016/j.net.2022.11.008

solidified radioactive waste specimen with no aggregate. Therefore, it is essential to conduct a quantified assessment for such solidified radioactive waste specimen.

In this study, we evaluated the effect of test conditions (H/D ratio, porosity, and loading rate) on the measured compressive strength of solidified radioactive waste. The manufactured specimen solidifies the mock sludge waste. Considering an increase in the diameter of the specimen increases the amount of secondary waste, the diameter of the specimen is fixed to 5 cm which is minimum diameter, and the effect of pore density and H/D ratio on the compressive strength is evaluated. Furthermore, the compressive strength test methods, ASTM C39 and KS F 2405, are compared to check the effect of loading rate on the compressive strength measurement. The significant effects of the test conditions on the compressive strength are analyzed through the statistical method. Finally, we suggest the compressive strength test method that measures its strength in the most reasonable and conservative way by analyzing the relationship between test conditions and compressive strength.

2. Methods

2.1. Preparation of cement solidified specimen

$Ca(OH)_2$ and CaF_2 were selected as mock sludge waste, which are the main precipitant and precipitate during the conversion process of UF_6, respectively. The mock sludge waste was prepared by mixing $Ca(OH)_2$ (ThermoFisher scientific, 95%) and CaF_2 (ThermoFisher scientific, 97%) in a 1:1 ratio with water. The content of the sludge waste inside the specimen was controlled by dividing the cement content into 40, 50, and 60 wt%, as shown in Table 1, to make specimens with compressive strengths close to the WAC (3.44 MPa). The solidification of the waste was performed by using type I cement (Ordinary Portland cement) produced by Asia Cement as binding material. Owing to the large volume of sludge compared with cement, a maximum 2 wt% of plasticizer was added during mixing to enhance workability. Finally, the cement solidified waste specimen was manufactured by using a mixing bowl clarified in KS L ISO 679 [15]. The mixing procedure is as follows:

(1) Mix water and cement at slow speed for 30 s
(2) Add sand (sludge in this study) and mix for 30 s
(3) Mix at fast speed for 30 s
(4) Stop mixing for 90 s and segregate the cement on the wall using scrapper for 15 s
(5) Mix at fast speed for 60 s

However, few cement solidified specimen are fractured after removing the mold of specimen. To maximize the amount of sludge, the aggregate which is generally added for cement mortar and cement is excluded. The low compressive strength of specimen and absence of well-graded aggregate unlike concrete and cement mortar are expected to be the reasons of damage on the specimen after the curing period. Therefore, damaged specimens after curing process are excluded from the micro-CT analysis and compressive strength experiment.

Table 1
Weight percentage of ingredients in the cement solidified specimen.

	Cement (wt.%)	Water (wt.%)	Sludge (wt.%)	Plasticizer (wt.%)
#1	40	21	39	1–2
#2	50	18	32	
#3	60	14	26	

2.2. Dimension of cement solidified specimen

In the WAC of Gyeongju repository and "NRC, technical position on waste form, rev1," solidified waste specimen should satisfy the H/D ratio of 2 and a minimum diameter of 50 mm. Additionally, a weighting factor is provided for specimens that do not satisfy the required H/D ratio in "NRC, technical position on waste form, rev 1." However, the compressive strength of cement solidified waste differs depending on the H/D ratio [11,12]; the H/D ratio of 200 L drum is less than 2. Therefore, as shown in Fig. 1, cement solidified specimen with an H/D ratio of 2 and 200 L drum (H/D = 1.44) were manufactured. Specimen with an H/D ratio of 2 satisfied the standard dimensions clarified in the WAC of Gyeongju LILW, i.e., a diameter and height of 5 cm and 10 cm, respectively. The specimen with an H/D ratio of 200 L drum were developed by varying the height of the specimen above the specified dimension, where the diameter was 5 cm and height was 7.15 cm.

2.3. Measurement of porosity of cement solidified specimen

The pore distribution of the specimens was examined by micro-computed tomography (micro-CT) using the Nikon XT H 320LC with 16-bit grayscale resolution before compressive strength measurements. The image of micro-CT has been analyzed using VG STUDIO, as shown in Fig. 2. The pores in the specimen are depicted in the image as the darkest parts, and the other materials have brighter shades depending on the material's permeability. For this reason, the micro-CT has been widely used in investigating the pore and aggregate distribution in concrete samples non-destructively [16–18]. The specimens simulating solidified radioactive waste in this study did not contain aggregates, so the two colors were mainly identified, indicating pores and mortals, respectively. The porosity and maximum pore size were primarily analyzed since they would be one of the most critical parameters affecting the cement and concrete compressive strength when the specimen is typically prepared [8,19,20]. Pore volume and total porosity were determined from the three-dimensional scanned image, and the size of the non-spherical pore was defined as the longest part.

2.4. Loading rate of compressive strength measurement

Generally, the measured compressive strength differs depending on the loading rate, that is, a higher loading rate causes higher measured compressive strength. Radioactive waste repositories in U.S. follows the loading rate of ASTM C39, which is 0.25 ± 0.05 MPa/s for cement solidified waste [14]. The WAC of Gyeongju LILW

Fig. 1. (a) Cement solidified specimen with an H/D ratio of 2 and (b) H/D ratio of 200 L drum (1.44).

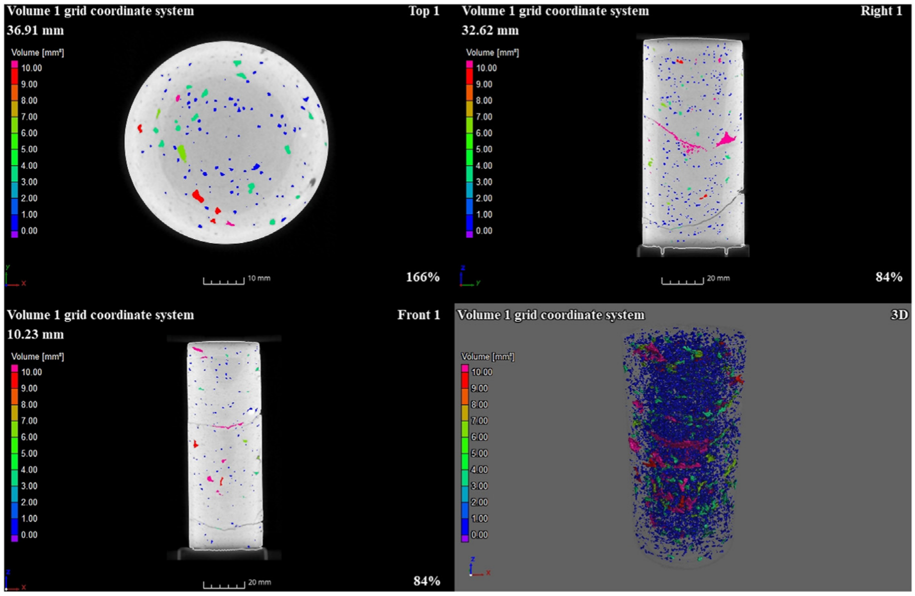

Fig. 2. Image of pores inside the cement solidified specimen by Microfocus 3D CT system.

repository follows the loading rate of KS F 2405, which is 0.6 MPa/s for cement solidified waste [21]. Therefore, the assessment that quantifies the effect of loading rate on the cement solidified waste should be developed and considered based on the aspect of regulation. In this study, the minimum loading rate of ASTM C39 of 0.2 MPa/s was set to derive the maximum difference in the loading rate between ASTM C39 and KS F 2405. The overall compressive strength measurements are performed using the AGX-50kNVD compression machine, considering the compressive strength of the specimen is expected to be less than 20 MPa. However, for the outlier specimen that has a compressive strength larger than 20 MPa, the compressive strength measurement is conducted using the KSU-HSO200 compression machine.

2.5. Statistical analyses of compressive strength

The result of the compressive strength test showed that a simple observation is insufficient to observe the significant effect of the test conditions on the compressive strength. Therefore, the effect of test conditions on compressive strength can be statistically analyzed using the SPSS statistical analysis program by reflecting a small number of specimens and large deviations in their strength value. Statistical analysis can be divided into two sections depending on the normality of the dataset: parametric and nonparametric statistical analysis. To check whether the compressive strength dataset satisfies normality, Shapiro's test was performed by the SPSS program. The results showed that the compressive strength dataset satisfied the normality; however, both parametric and nonparametric statistical analyses were performed considering the amount of compressive strength data per specimen size and significantly small test condition.

To check the effect of loading rate on compressive strength, a statistical analysis was performed on the compressive strength dataset with the same H/D ratio and cement content. Furthermore, to examine the effect of H/D ratio, the statistical analysis was conducted for the compressive strength dataset with the same loading rate and cement content. Both parametric and nonparametric statistical analyses were performed for the compressive strength dataset with the same loading rate, H/D ratio, and cement content when analyzing the correlation between compressive strength and porosity. During the analyses, the two-sided test was applied, and the level of significance was set to 0.05.

2.5.1. Student's t-test

Student's t-test is a statistical hypothesis test that follows the Student's t-distribution under the null hypothesis [22]. Two sample t-tests were conducted to check whether the average population of two independent groups were different and both groups satisfied normality, homogeneity of variance, and independency. The normality of the sample was checked by performing Shapiro's test or Kolmogorov-Smirnov test, whereas the homogeneity of variance was checked by conducting Levene's test. By assuming the normality and homogeneity of variance of two independent sample groups, t-value is expressed as:

$$t = \frac{\overline{X}_1 - \overline{X}_2}{s_{(1+2)}\sqrt{\frac{1}{n_1} + \frac{1}{n_2}}}\ (df = n_1 + n_2 - 2) \tag{1}$$

where n_1 and n_2 are the sample size, $\overline{X}_1$ and $\overline{X}_2$ are sample mean of groups 1 and 2, respectively, and s is the standard deviation of both groups. The integrated variance of sample is applied as the population variance of the distribution of two independent sample mean difference is unknown, given as:

$$s^2_{(1+2)} = \frac{(n_1 - 1)s_1^2 + (n_2 - 1)s_2^2}{n_1 + n_2 - 2} \tag{2}$$

If the homogeneity variance of two sample groups cannot be assumed, the t-value can be expressed as:

$$t = \frac{\overline{X}_1 - \overline{X}_2}{\sqrt{\frac{s_1^2}{n_1} + \frac{s_2^2}{n_2}}} \tag{3}$$

The p-value is calculated by the t-value derived from Eq (3), and the adoption of the null hypothesis is decided by comparing the p-value to a significant level. A two-sided test was conducted to check whether the two sample groups are from the same population.

2.5.2. Mann-Whitney U test

The Mann-Whitney U (MWU) test, a representative nonparametric statistical test that checks whether two independent sample groups are from same population [23], was applied when the number of samples was small enough and normality was not satisfied. The null hypothesis in the MWU test assumes that two sample groups are from the same population, whereas the alternative hypothesis assumes both groups are from different populations. Two groups with n_x and n_y number of samples were compared individually by applying a rank after merging two groups and sort in ascending order. The U value of the MWU test is expressed as:

$$\mathrm{U_x} = n_x n_y + \left(\frac{(n_x(n_x+1))}{2}\right) - R_x \tag{4}$$

$$\mathrm{U}_y = n_x n_y + \left(\frac{(n_y(n_y+1))}{2}\right) - R_y \tag{5}$$

where n_x and n_y are the sample sizes in two independent groups and R_x and R_y are the ranks applied to two independent groups.

The p-value was derived by substituting the smaller U value from the U values from Eqs (4) and (5) to the Mann-Whitney table, whereas the adoption of the null hypothesis was decided by comparing the p-value to the significant level.

$$\mathrm{H_0}\ \mathit{if}\ p\ \mathit{of}\ \min(U_x, U_y) < \alpha\ \mathit{threshold} \tag{6}$$

If the sample size in each group is larger than 8, the below equation can be applied:

$$\mu_{\mathrm{U}} = \frac{n_x n_y}{2} = \frac{U_x + U_y}{2}\ \mathit{and}\ \sigma_U = \sqrt{\frac{(n_x n_y)(N+1)}{12}} \tag{7}$$

where N is sum of n_x and n_y, μ is sum of U variance, and σ is the standard deviation.

Additionally, when the sample size was larger than 8, the distribution of the sample group was close to the normal distribution and z-value, expressed as:

$$\mathrm{z} = \frac{\left(\mathrm{U} - \left(\frac{\mathrm{n_x} n_y}{2}\right)\right)}{\sigma U} \tag{8}$$

The adoption of the null hypothesis was decided by applying the z-value derived from Eq (8).

2.5.3. Pearson's correlation coefficient

The Pearson's correlation coefficient is a value that quantifies the linear correlation between two parameters [24,25], which should be interval or continuous and satisfy normality to perform the Pearson's correlation test. The Pearson's coefficient $\mathrm{r_p}$ is given as:

$$\mathrm{r_p} = \frac{\sum_{i=1}^{n}(x_i - \overline{x})(y_i - \overline{y})}{\sqrt{\sum_{i=1}^{n}(x_i - \overline{x})^2}\sqrt{\sum_{i=1}^{n}(y_i - \overline{y})^2}} \tag{9}$$

where $\overline{x}$ and $\overline{y}$ are the average of x_i and y_i, respectively, and n is the number of samples. The Pearson's coefficient ranges between −1 and +1, and the values close to +1, −1, and 0 denote strong positive correlation, strong negative correlation, and no correlation, respectively. The p-value can be derived, and if it is lesser than the level of significance, the correlation between two parameters is equivalent to the correlation coefficient obtained.

2.5.4. Spearman's rho correlation coefficient

Spearman's rho correlation coefficient is a nonparametric method for calculating the correlation coefficient by using rank between two parameters [26,27]. Spearman's correlation is applied when a sample is ordinal or continuous and the sample group does not satisfy normality. Spearman's correlation coefficient of two parameters x and y is given as:

$$\rho = 1 - \frac{6\sum_{i=1}^{n} d_i^2}{n(n^2-1)} \tag{10}$$

$$\mathrm{d_i} = \mathrm{x_i} - y_i \tag{11}$$

where ρ is the Spearman's correlation coefficient, n is the number of samples, and d_i is the difference between the ranks of X and Y. The Spearman's correlation coefficient ranges from −1 to +1.

3. Results

3.1. Measured compressive strengths and porosity

Fig. 3 shows the measured compressive strength of the cement solidified specimen with three different cement contents. The results of the compressive strength measurement, average, and standard error with 95% confidential level are shown in Table 2. The porosity values measured by Micro 3D CT are shown in Table 3. The first three maximum porosities are 5.89, 5.79, and 5.65%, whose compressive strengths are 9.34, 13.95, and 7.71 MPa, respectively. The last three are 0.03, 0.17, and 0.71% with 5.95, 14.26, and 18.02 MPa, respectively. Additionally, the non-existence of aggregate was observed through the color difference shown in the CT image.

3.2. Effect of H/D ratio and loading rate on compressive strength

Student's t-test and MWU test were performed to analyze the effect of loading rate and H/D ratio on the compressive strength of cement solidified waste. Levene's test was conducted to evaluate the homogeneity of variance during the Student's t-test. The level of significance (α) in the overall study was set to a general value, 0.05. Although the p-values from the Student's t-test and MWU test were different, the same result was derived. Table 4 shows the results of the Student's t-test and MWU test examining the effect of loading rate on the compressive strength, while the H/D ratio is fixed. Furthermore, the significant effect of loading rate on the compressive strength was evaluated for the cement solidified waste with cement content #1 and #2 when the H/D ratio is two. Additionally, the heterogeneity of variance was found when the significant effect of test condition was evaluated. In other cases, the insignificant effect of loading rate on the compressive strength and homogeneity of variance were evaluated. Table 5 shows the results of the test that evaluates the effect of H/D ratio on the compressive strength when the loading rate was fixed. Solidified waste with cement contents #1 and #2 only showed the significant effect of H/D ratio when the loading rate was fixed to ASTM C39. For other cases, no significant effect of H/D ratio was found.

3.3. Correlation analysis between porosity and compressive strength

The correlation between porosity and compressive strength was

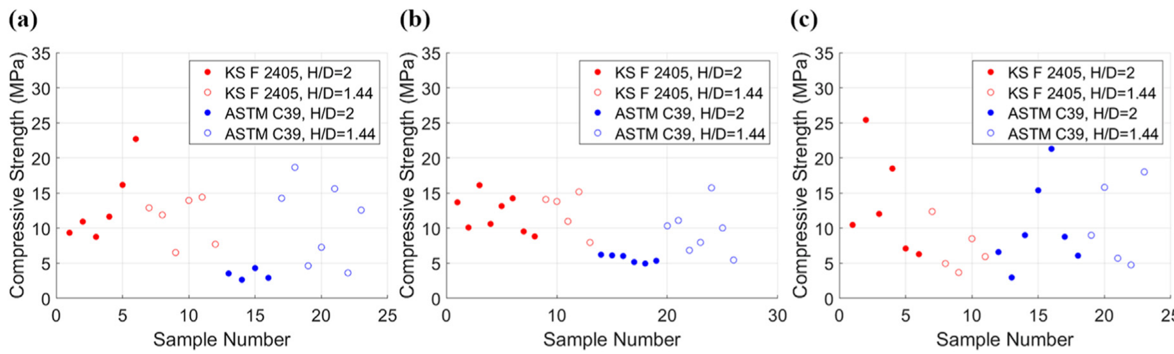

Fig. 3. Measured compressive strengths of cement solidified specimen with cement content (a) #1 (40 wt%), (b) #2 (50 wt%), and (c) #3 (60 wt%).

Table 2
Measured compressive strength according to the test conditions.

	KS F 2405 (H/D = 2)			ASTM C39 (H/D = 2)			KS F 2405 (H/D = 1.44)			ASTM C39 (H/D = 1.44)		
	#1	**#2**	**#3**	**#1**	**#2**	**#3**	**#1**	**#2**	**#3**	**#1**	**#2**	**#3**
M[a] (MPa)	10.93	10.09	10.46	2.65	4.96	15.38	11.88	10.97	12.37	12.57	10.02	15.81
	11.64	10.6	12.03	2.92	5.17	2.97	12.89	13.8	3.69	14.26	10.32	18.02
	16.16	13.14	18.49	3.54	5.35	6.08	13.95	14.09	4.96	15.26	11.1	4.76
	8.76	13.68	6.29	4.31	6.03	6.59	14.41	15.17	5.95	18.66	15.74	5.71
	9.34	14.25	7.09		6.12	8.77	6.52	7.96	8.5	3.63	5.46	8.99
	22.7	16.12	25.42		6.22	9	7.71			4.64	6.85	
		8.82				21.3				7.27	7.97	
		9.53										
Avg[b] (MPa)	13.25	12.03	13.30	3.36	5.64	10.01	11.23	12.40	7.09	10.95	9.64	10.66
SE[c] (MPa)	2.17	0.93	3.01	1.36	1.31	1.54	0.37	0.22	2.37	2.19	1.27	2.67

[a] Measured compressive strength.
[b] Average.
[c] Standard Error.

Table 3
Porosity measurement results of the cement solidified specimen.

	KS F 2405 (H/D = 2)			ASTM C39 (H/D = 2)			KS F 2405 (H/D = 1.44)			ASTM C39 (H/D = 1.44)		
	#1	**#2**	**#3**	**#1**	**#2**	**#3**	**#1**	**#2**	**#3**	**#1**	**#2**	**#3**
M[a] ($\times 10^{-2}$)	4.03	4.04	2.83	2.09	3.40	.00	5.07	4.59	0.70	2.43	1.69	1.37
	3.85	4.66	3.61	2.78	2.98	.32	4.49	4.52	0.69	0.17	1.43	0.71
	4.34	5.01	3.17	2.77	3.22	.96	5.79	4.23	0.82	2.61	1.21	0.83
	4.31	4.37	3.76	3.14	3.05	.35	3.67	4.21	0.03	4.33	1.84	0.77
	5.89	4.52	2.65		3.04	.11	5.50	4.07	1.37	2.12	1.55	.89
	3.98	4.29	3.65		3.46	3.40	5.65			2.24	1.25	
		5.31				3.23				2.37	1.27	
		5.13										

[a] Measured porosity.

Table 4
Results of the Student's t-test and the Mann-Whitney *U* test between the loading rate and compressive strength while H/D ratio is fixed.

		H/D = 2			H/D = 1.44		
		#1	#2	#3	#1	#2	#3
p-value	**Levene**	0.039	0.001	0.630	0.064	0.911	0.090
	***t*-test**	0.006	0.000	0.411	0.920	0.171	0.281
	MWU[a]	0.010	0.001	0.366	1.000	0.268	0.421

[a] Mann-Whitney *U* test.

examined by performing the Pearson's correlation test and the Spearman's correlation test, which are parametric and nonparametric methods, respectively. Table 6 summarizes the results, which showed no correlation between the porosity and compressive strength. In the Spearman's correlation test, the specimen with cement content #3 was tested under the loading rate of ASTM C39 and H/D ratio of 2 showed p-value smaller than the level of significance where the correlation coefficient was 0.786.

3.4. *Correlation analysis between maximum pore volume and compressive strength*

The correlation between the maximum pore volume and the compressive strength was checked through the Pearson's

Table 5
Results of Student's t-test and Mann-Whitney *U* test between H/D ratio and compressive strength while loading rate is fixed.

		KS F 2405			ASTM C39		
		#1	#2	#3	#1	#2	#3
p-value	**Levene**	0.310	0.903	0.126	0.004	0.039	0.895
	***t*-test**	0.446	0.953	0.119	0.013	0.020	0.862
	MWU[a]	0.818	0.724	0.126	0.012	0.008	.000

[a] Mann-Whitney *U* test.

Table 6
Correlation analysis results between porosity and compressive strength.

		KS F 2405 (H/D = 2)			ASTM C39 (H/D = 2)			KS F 2405 (H/D = 1.44)			ASTM C39 (H/D = 1.44)		
		#1	#2	#3	#1	#2	#3	#1	#2	#3	#1	#2	#3
p-value	**P**[a]	0.446	0.224	0.554	0.148	0.945	0.170	0.252	0.733	0.715	0.548	0.232	0.580
	S[b]	0.397	0.120	0.872	0.200	0.704	0.036	0.468	1.000	0.624	0.119	0.702	0.873

[a] Pearson's correlation.
[b] Spearman's correlation.

correlation test and the Spearman's correlation test. Table 7 summarizes the analyses results where no correlation between the maximum pore volume and the compressive strength was observed. All the p-values of specimen showed lower value compared to the level of significance (see Table 7).

4. Discussion

As shown in Fig. 3, the difference in the compressive strength due to cement content was hard to observe owing to the large variance in cement specimen and different test conditions. Therefore, it was not possible to evaluate the effect of the test conditions, height-to-diameter (H/D) ratio, loading rate, and porosity, in the statistical analyses. Through the Student's t-test and the MWU test, the effect of loading rate and H/D ratio on the compressive strength of cement solidified specimen was examined. Although the p-values derived from these tests were different, the results were the same. With the specimen with an H/D ratio of 2 and cement content #1(40 wt%) and #2(50 wt%), the significant effect of the loading rate and compressive strength was evaluated (ASTM C39: 0.2 MPa/s & KS F 2405: 0.6 MPa/s). Additionally, the significant effect of the H/D ratio on the compressive strength was evaluated when the loading rate was ASTM C39 and cement content was #1 and #2. The loading rate of KS F 2405 showed no significant effect in terms of the H/D ratio on the compressive strength. Therefore, to effectively measure the compressive strength of cement solidified waste reflecting its cement content, the H/D ratio should be 2 and the loading rate should be adequately low.

Generally, the compressive strength of a concrete specimen is affected by the porosity with negative correlation [8,19,20]. However, unlike concrete specimen, cement solidified radioactive waste does not contain aggregates and has lower compressive strength owing to large amounts of waste. Therefore, the correlation between porosity and compressive strength of cement solidified waste specimen was tested, and the Pearson's correlation test showed no correlation between them. The Spearman's correlation test demonstrated that only one specimen showed a correlation between compressive strength and porosity. The specimen had cement content of #3(60 wt%) and H/D ratio of 2 with the loading rate of ASTM C39. However, all specimens except one showed no correlation between porosity and compressive strength. Therefore, most of the compressive strength of cement solidified waste had no correlation to the porosity.

Table 8
Example of checking the range of compressive strength of three cored solidified waste reflecting the standard error.

Unit (MPa)	Case #1	Case #2	Case #3
Specimen #1	3.5	3.5	3.5
Specimen #2	4	7	4
Specimen #3	5	10.5	10
Average strength	4.17	7	5.83
Standard error	±0.62	±2.86	±2.95
Range of strength	3.55–4,79	4.14–9.85	2.88–8.79
Satisfying criteria?	**O**	**O**	**X**

Excluding the porosity of the specimen, the maximum pore volume of the specimen can be one of the main factors that is critical to the compressive strength of the specimen as it is equal to the size of the defect inside the specimen. Some specimen showed significantly large pore volume where volume is larger than 100 m^3. However, no correlation between maximum pore size and compressive strength was found through the Pearson's and Spearman's correlation analyses for all specimens where all the p-values are less than 5%. Therefore, the maximum pore size of the specimen is expected to be not correlated to the compressive strength of the cement solidified specimen like the porosity.

In Gyeongju LILW repository, the average compressive strength of three core specimen from waste drum must satisfy the minimum

Table 7
Correlation analysis results between maximum pore volume and compressive strength.

		KS F 2405 (H/D = 2)			ASTM C39 (H/D = 2)			KS F 2405 (H/D = 1.44)			ASTM C39 (H/D = 1.44)		
		#1	#2	#3	#1	#2	#3	#1	#2	#3	#1	#2	#3
p-value	**P**[a]	0.430	0.671	0.299	.114	0.501	0.145	0.521	0.080	0.395	0.142	0.374	0.277
	S[b]	0.787	0.911	0.156	0.200	0.072	0.180	0.111	0.285	0.285	0.180	0.939	0.391

[a] Pearson's correlation.
[b] Spearman's correlation.

compressive strength of 3.44 MPa. The compressive strength test method was KS F 2405 for the solidified waste with a minimum diameter of 50 mm and an H/D ratio of 2. However, in "NRC, technical position on waste form, rev 1," the compressive strength test method followed ASTM C39 (0.25 ± 0.05 MPa/s), where the loading rate was less than the half of that of KS F 2405 (0.6 MPa/s) with the same compressive strength criterion. From the result of the study, the overall measured compressive strength of cement solidified waste specimen under loading rate of KS F 2405 showed a larger value compared to the results of ASTM C39, regardless of the cement content. Furthermore, measuring the compressive strength of cement solidified specimen with a large loading rate could overestimate the compressive strength and may not reflect its strength depending on the cement content. Therefore, the test method with lower loading rate is suitable for measuring the compressive strength of cement solidified specimen conservatively reflecting its cement content.

The concrete specimen with less height-to-diameter ratio commonly has higher compressive strength which corresponds to the results of this study. For this reason, ASTM C39 suggests using a weighting factor when the height-to-diameter ratio is less than two. The result of this study also corresponds: the ratio of 1.4 has higher compressive strength than that of the two. In the aspect of safety securement of the radioactive waste, it would be more conservative to use the ratio of two for the compressive test. Therefore, one must apply a lower loading rate than the KS F 2405 to the specimen with a fixed H/D ratio of 2 to measure the compressive strength of specimen conservatively.

To maximize the amount of radioactive waste in the solidified waste, the addition of the aggregate was ignored unlike the cement mortar and concrete. By adding aggregate, the dimensional stability, compressive strength, and prevention of shrinkage of the specimen can be secured. Nevertheless, those specimen with well-graded aggregate with specified mixture recipe cannot avoid the significant deviation of strength. Concrete can show the compressive strength in the range of 47.2 MPa–69.2 MPa where maximum difference is 22 MPa [28]. The cement mortar applied as brick joint showed the strength in the range of 1.55 MPa–3.26 MPa where maximum difference is 1.71 MPa [29]. Therefore, the large deviation of compressive strength for the specimen without aggregate and mixture recipe is expected to be inevitable and the possibility of large strength deviation of cement solidified radioactive waste should be considered in the radioactive waste repository.

In Gyeongju LILW repository, the compressive strength criterion was compared to the average compressive strength of a three cored specimen. However, the compressive strengths of cement solidified specimen showed large variance, then the average value is hard to be a representative of the actual strength of the waste. Additionally, the average compressive strength satisfied the criterion owing to its large variance, and some part of the solidified waste exhibited lower compressive strength compared with the criterion, as shown in Table 8. For case #3, all the compressive strength and average values satisfied the criterion; however, there was a possibility of lower compressive strength for the entire solidified waste, reflecting its large variance. Therefore, applying the standard error, which compensated the large variance in measured compressive strengths to the criterion, was suggested in this study.

Cement solidified specimens were made using mock sludge, where the main sludge waste ($(Ca(OH))_2$ and $CaF_{2)}$ from the UF_6 conversion process were mixed. However, several types of radioactive waste exist, and solidified waste containing other types of waste may show different results. Based on the study results, the compressive strength of cement solidified specimen exhibited a large variance, which was significantly affected by the working environment. In addition, other solidifying methods, including vitrification and polymer solidification, are possible, and cement solidification might not be conducted in the future when there are changes in the WAC of repository [30–32]. Therefore, in future works, compressive strength measurements should be performed according to the test method for the different types of solidifying media.

5. Conclusion

In this study, compressive strength measurements of cement specimens, which solidified sludge waste with various cement content, were performed by varying the height-to-diameter ratio and loading rate after porosity analysis. The compressive strength reflecting the low content cement in the specimen was measured through parametric and nonparametric analyses when the loading rate was low and the H/D ratio was equal to 2. However, no effect of naturally generated pores inside the specimen during curing period on the compressive strength was checked. Therefore, to avoid overestimation of the cement solidified radioactive waste, one must apply a lower loading rate during the compressive strength test in the radioactive waste repository. However, in future works, similar studies should be conducted for cement solidified waste with different types of waste and specimen with different solidifying media.

Funding

This work was supported by the Nuclear Safety Research Program through the Korea Foundation of Nuclear Safety (KoFONS), granted financial resources from the Nuclear Safety and Security Commission (NSSC), Republic of Korea (No. 1903005 & No. 2203028).

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.

References

[1] International Atomic Energy Agency, Underground Disposal of Radioactive Waste: Basic Guidance, IAEA Safety Series No. 54, International Arts and Entertainment Alliance, Vienna, 1981.
[2] M. Fuhrmann, R.F. Pietrzak, E.M. Franz, J.H. Heiser III, P. Colombo, Optimization of the Factors that Accelerate Leaching (No. BNL-52204), Brookhaven National Laboratory, Upton, New York, 1989.
[3] R.F. Pietrzak, M. Fuhrmann, E.M. Franz, J. Heiser III, P. Colombo, Accelerated Leach Testing of Radionuclides from Solidified Low-Level Waste, 29, American Chemical Society, Division of Environmental Chemistry, Preprints, 1989. CONF-8904178–).
[4] Korea Radioactive Waste Agency (KORAD). SAfety Analysis Report for Low- and Intermediate-Level Radioactive Waste Disposal Facility.
[5] U.S. Nrc, Technical Position on Waste Form, First Revision. Final Waste Classification and Waste Form Technical Po-Sition Papcrs, United States Nuclear Regulatory Commission, Low-Levcl Waste Licensing Branch, Washington, District of Columbia, 1991.
[6] A. Abdullah, Effects of specimen sizes and loading rates on compressive strength of concrete, Mater. Today Proc. 46 (2021) 1783–1786, https://doi.org/10.1016/j.matpr.2020.07.613.
[7] H.C. Fu, M.A. Erki, M. Seckin, Review of effects of loading rate on reinforced concrete, J. Struct. Eng. 117 (1991) 3660–3679, https://doi.org/10.1061/(ASCE)0733-9445(1991)117:12(3660).
[8] P.K. Mehta, P.J. Monteiro, Concrete: Microstructure, Properties, and Materials, McGraw-Hill Education, 2014.
[9] J.I. Sim, K.H. Yang, H.Y. Kim, B.J. Choi, Size and shape effects on compressive strength of lightweight concrete, Construct. Build. Mater. 38 (2013) 854–864, https://doi.org/10.1016/j.conbuildmat.2012.09.073.
[10] S.T. Yi, E.I. Yang, J.C. Choi, Effect of specimen sizes, specimen shapes, and placement directions on compressive strength of concrete, Nucl. Eng. Des. 236 (2006) 115–127, https://doi.org/10.1016/j.nucengdes.2005.08.004.
[11] F.M. Bartlett, J.G. MacGreggor, Effect of core length-to-diameter ratio on

concrete core strengths, Materials. Journal. 91 (1994) 339–348.
[12] M. Li, H. Hao, Y. Shi, Y. Hao, Specimen shape and size effects on the concrete compressive strength under static and dynamic tests, Construct. Build. Mater. 161 (2018) 84–93, https://doi.org/10.1016/j.conbuildmat.2017.11.069.
[13] K.H. Kim, Y.G. Ryu, T.K. Kim, Comparison of Various Standard Test Methods for Characterization of Radioactive Waste Forms, Korea Atomic Energy Research Institute, 2008. No. KAERI/TR–3695/2008.
[14] ASTM C39/C39M, Standard Test Method for Compressive Strength of Cylindrical Concrete Specimens, ASTM International, West Conshohocken, 2016.
[15] Korean Standards Association, L. Ks, ISO 679 Methods of Testing Cements-Determination of Strength, KSA, Korea, 2006.
[16] I. Marzec, J. Tejchman, Fracture evolution in concrete compressive fatigue experiments based on X-ray micro-CT images, Int. J. Fatig. 122 (2019) 256–272.
[17] J.S. Kim, S.Y. Chung, T.S. Han, D. Stephan, M. Abd Elrahman, Correlation between microstructural characteristics from micro-CT of f oamed concrete and mechanical behaviors evaluated by experiments and simulations, Cement Concr. Compos. 112 (2020), 103657.
[18] S.Y. Chung, C. Lehmann, M. Abd Elrahman, D. Stephan, Pore characteristics and their effects on the material properties of foamed concrete evaluated using micro-CT images and numerical approaches, Appl. Sci. 7 (6) (2017) 550.
[19] X. Chen, S. Wu, J. Zhou, Influence of porosity on compressive and tensile strength of cement mortar, Construct. Build. Mater. 40 (2013) 869–874, https://doi.org/10.1016/j.conbuildmat.2012.11.072.
[20] C. Lian, Y. Zhuge, S. Beecham, The relationship between porosity and strength for porous concrete, Construct. Build. Mater. 25 (2011) 4294–4298, https://doi.org/10.1016/j.conbuildmat.2011.05.005.
[21] F.2405 Ks, Standard Test Method for Compressive Strength of Concrete, KSSN, 2015.
[22] T.K. Kim, , t test as a parametric statistic, Korean J. Anesthesiol. 68 (2015) 540–546, https://doi.org/10.4097/kjae.2015.68.6.540.
[23] N. Nachar, The mann-whitney U: a test for assessing whether two independent samples come from the same distribution U: a test for assessing whether two independent samples come from the same distribution, Tutor. Quant. Methods Psychol. 4 (2008) 13–20, https://doi.org/10.20982/tqmp.04.1.p013.
[24] J. Benesty, J. Chen, Y. Huang, I. Cohen, Pearson correlation coefficient, in: Springer Topics in Signal Processing, Springer, Berlin, Heidelberg, 2009, pp. 1–4, https://doi.org/10.1007/978-3-642-00296-0_5.
[25] A.M. Neto, A.C. Victorino, I. Fantoni, D.E. Zampieri, J.V. Ferreira, D.A. Lima, Image processing using Pearson's correlation coefficient: applications on autonomous robotics, in: 2013 13th International Conference on Autonomous Robot Systems, IEEE Publications, 2013, pp. 1–6.
[26] D. Liu, S.Y. Cho, D.M. Sun, Z.D. Qiu, A Spearman correlation coefficient ranking for matching-score fusion on speaker recognition, in: TENCON 2010-2010 IEEE Region 10 Conference, IEEE Publications, 2010, pp. 736–741.
[27] C. Xiao, J. Ye, R.M. Esteves, C. Rong, Using Spearman's correlation coefficients for exploratory data analysis on big dataset, Concurrency Comput. Pract. Ex. 28 (2016) 3866–3878, https://doi.org/10.1002/cpe.3745.
[28] K.B. Rao, S.M. Ibrahim, The standard deviation in cube strength results of self-compacting concrete of m40 grade using quarry dust as fine aggregate and fly ash powder as filler, Int. J. Civ. Eng. Technol. 8 (10) (2017) 1695–1703.
[29] D. Łątka, P. Matysek, Assessment of the compressive strength of lime mortar in the joints of brick walls-case study, in: MATEC Web of Conferences, vol. 163, EDP Sciences, 2018, 02006.
[30] M.G. Cowgill, A Comparison of Solidification Media for the Stabilization of Low-Level Radioactive Wastes (No. BNL–52304), Brookhaven National Laboratory, 1991.
[31] J.K. Park, M.J. Song, Feasibility study on vitrification of low-and intermediate-level radioactive waste from pressurized water reactors, Waste Manag. 18 (1998) 157–167, https://doi.org/10.1016/S0956-053X(98)00017-8.
[32] C.C. Tzeng, Y.Y. Kuo, T.F. Huang, D.L. Lin, Y.J. Yu, Treatment of radioactive wastes by plasma incineration and vitrification for final disposal, J. Hazard Mater. 58 (1998) 207–220, https://doi.org/10.1016/S0304-3894(97)00132-5.