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Preparation and Properties of Sustainable Concrete Using Activated Sludge of Industrial By-Products

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Abstract: Industrial sludge byproducts contain CaO, SiO_2, Al_2O_3, etc. When industrial sludge is used in ready-mixed concrete, the performance of the concrete can be enhanced due to the hydration reaction. In the present study, activated sludge was used to prepare ready-mixed concrete, and its durability performance was evaluated. Once the activated sludge was used, the durability of the concrete improved. Therefore, it is suggested that activated sludge can be used in concrete mix as an admixture.

Keywords: activated sludge; industrial byproduct; desulfurization gypsum; recycled water; ready-mixed concrete; durability of concrete

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1. Introduction

The ready-mixed concrete industry has been popular in the construction sector since the 1960s in the United States and Japan. Therefore, a number of ready-mixed concrete industries have been established, and the production of concrete has increased proportionally. In the United States, approximately 4000 ready-mixed concrete companies were established in the 1960s. Until this period, about 60% of cement was used for the production of ready-mixed concrete [1]. As time has gone by, the number of companies has increased, as has the production of ready-mixed concrete [2]. However, in Japan, the ready-mixed concrete industry was introduced in 1949, and the construction industry was revitalized through the restoration project and economic development after World War II [1]. In 2019, the production of ready-mixed concrete reached around 81 million m^3 [3]. South Korea introduced the production of ready-mixed concrete in 1965 through the development of a national construction industry. Currently, in South Korea, about 1083 companies are producing 147 million m^3 of ready-mixed concrete annually [4]. In addition, as the number of companies and production has increased, the amount of recycled water from ready-mixed concrete plants has also increased. Shin et al. studied the recycled water from ready-mixed concrete and found that until 2002, about 0.2 tons per 1 m^3 of concrete was used [5]. Moreover, during the washing of mixer trucks, drums, and batching plants after concrete production, a large amount of drainage water was used, which lowered the groundwater level [6]. This drainage water is considered wastewater and contains supernatants, fine aggregates, coarse aggregates, and sludge [7]. However, this water can be recycled and used in the mixing of concrete again.

In ready-mixed concrete production, the generation of recycled water is inevitable and needs attention for its efficient use [8]. In Europe and the USA, it is prohibited to discharge recycled water if its pH is lower than 11.5 [9,10]. However, this water can be used after treatment, keeping in mind the environment, cost, and energy [11–13]. After treatment, this water can be discharged or disposed of in soil by maintaining its quality, thus avoiding pollution [14,15]. Since various regulations on environmental pollution have been enacted, it is required to maintain the quality of the byproducts of industries using treatment technology. It was suggested by the Recycled Water Research Committee of the Japan Concrete Engineering Association in 1975 that the sludge of ready-mixed concrete can be used in concrete [16].

Lee et al. utilized less than 4% sludge of ready-mixed concrete and produced durable concrete [17]. Han et al. used different amounts of recycled water with different compositions to make durable concrete [18,19]. They recommended the use of either recycled water of ready-mixed concrete or chemical admixtures to prepare concrete. Kim et al. suggested that when 0.15% of a chemical admixture was used with recycled water, the properties of concrete were improved [20]. Lee et al. suggested using a retardant with an admixture to prepare durable concrete with recycled water [21,22]. Chatveera et al. reported that when sludge water was used to replace normal water for the production of concrete, the slump values decreased, and the compressive strength was found to be from 85 to 94% compared to normal concrete [23]. Sekolu and Dawneerangen reported that when 30, 50, and 100% tap water was used compared to recycled water, the compressive strength of mortar increased by 8% [24]. The use of recycled water from ready-mixed concrete can be utilized in concrete, but it slightly changes the properties [14,15]. Han et al. reported that as the amount of sludge solids increased, the amount of slump and air content decreased [7]. In general, it is suggested that if recycled water from ready-mixed concrete is used in concrete, the properties of concrete are decreased due to the solid content present in recycled water compared to tap water. However, it is recommended that instead of recycled water from ready-mixed concrete, a supernatant can be used in concrete. Moreover, KS F 4009 [25] and ASTM C94/C94 M-20 [26] recommended a procedure for making concrete using recycled water from ready-mixed concrete. If the conditions are suitable, as recommended in the standard, then only a limited amount of supernatants can be used, and sludge must be discarded [27].

Moreover, there is a requirement to reduce the cost of the process to make recycled water usable in concrete by taking into account environmental protection [28,29]. Therefore, in the present study, we took desulfurized gypsum (an industrial byproduct containing CaO) [30,31] to prepare activated sludge, which can accelerate the hydration reaction of concrete using recycled water at a high pH. We recommend that if recycled water is used in the form of activated sludge, then the cost of concrete production, environmental load, and CO_2 generation from cement industries can be decreased, and concrete can be considered eco-friendly [32–35].

2. Experiment Outline

The activated sludge was prepared using desulfurized gypsum (an industrial by-product). Table 1 shows the chemical composition of activated sludge measured by X-ray fluorescence (XRF). Activated sludge contains a high amount of CaO and SO_3 as well as a nominal amount of other oxides. The loss on ignition (LOI) is found to be highly attributed to the industrial by-products.

Table 1. Chemical composition of the activated sludge.

Flue Gas Desulfurization Gypsum	Oxides Content (Wt%)								
	CaO	SiO_2	Al_2O_3	SO_3	Fe_2O_3	MgO	Na_2O	K_2O	LOI
	60.5	4.78	0.86	25.6	0.46	0.81	0.04	0.25	6.50

2.1. Preparation of Activated Sludge and Cement Matrix

In the present study, recycled water was used to prepare the concrete [36]. Activated sludge was prepared by replacing 5 wt.% cement with desulfurized gypsum and recycled water [12,36].

The concrete specimen was prepared using Type 1 Portland cement according to ASTM C 150 [37]. The density and fineness of the cement was 3.15 g/cm^3 and 3200 cm^2/g, respectively. The concrete specimens were prepared according to ASTM C 109 [38].

2.2. Preparation of the Concrete

The concrete specimens were prepared by mixing 25 mm crushed coarse aggregate, sea sand (fine aggregate), fly ash, and blast furnace slag. The density of fly ash and blast furnace slag was 2.22 g/cm^3 and 2.90 g/cm^3, while fineness was 3317 cm^3/g and 4450 cm^3/g, respectively. A polycarboxylic acid-based superplasticizer (SP) was used to control the workability of the concrete. Table 2 shows the chemical composition of cement, blast furnace slag, and fly ash measured by XRF.

The water to binder (W/B) and fine aggregate (S/a) ratio was fixed at 50.8% and 49.5%, respectively, for the preparation of concrete mix to obtain 24 MPa compressive strength, 150 mm slump value, and 4.5% air content. There were three types of concrete mixes chosen. The first type of mix was plain concrete with no other admixture added (Table 3). In the second type of concrete mix, 10, 20, and 30% recycled water was used with respect to water (Table 3), while in third type of the concrete, 5% desulfurized gypsum was used to replace the cement along with recycled water (Table 3). In the specimen ID, the first letter indicates recycled water, the second number indicates the percentage of recycled water, and the third number indicates the percentage of desulfurized gypsum.

The concrete was mixed by a pen type mixer (HEUNGJIN, HJ-3369, Incheon, South Korea). Initially, aggregate and binder were mixed for 30 s at 20 rpm, and thereafter water was added at 30 rpm for 60 s. Once aggregates and binder were mixed properly, the admixture was added at 40 rpm for 90 s. After complete mixing of the concrete, it was poured into the mold. The curing was carried out in the air for 1 day at 20 ± 2 °C, and thereafter it was kept in water for 28 days curing.

Table 2. Chemical composition of cement, fly ash, and blast-furnace slag.

Oxides	Content (Wt.%)		
	Cement	**Fly Ash**	**Blast-Furnace Slag**
CaO	62.0	3.0	41.7
SiO_2	20.8	56.4	31.7
Al_2O_3	6.3	23.4	14.5
SO_3	2.2	0.4	2.1
Fe_2O_3	3.2	7.9	0.7
MgO	3.3	1.5	5.4
LOI	1.3	1.2	2.6

Table 3. Mix proportion of concrete.

Specimen ID	W/B * (%)	S/a * (%)	Kg/m^3								SP * (%)	AE * (%)
			C *	W *	FA *	BS *	RW *	DG *	G *	S *		
Plain	50.8	49.5	253	172	51	34	-	-	846	934	C× 0.5%	C× 0.1%
R-1-0			253				17.2	-				
R-2-0			253				34.4					
R-3-0			253				51.6	-				
R-1-5			240.35				17.2	12.65				
R-2-5			240.35				34.4	12.65				
R-3-5			240.35				51.6	12.65				

* W/B: water/binder ratio, S/a: fine aggregate ratio, W: water, C: cement, FA: fly ash, BS: blast-furnace slag RW: recycled water, DG: desulfurization gypsum S: fine aggregate, G: coarse aggregate, SP (superplasticizer): high range water reducing agent, AE: air entraining agent.

2.3. *Experiment Methods*

2.3.1. Compressive Strength

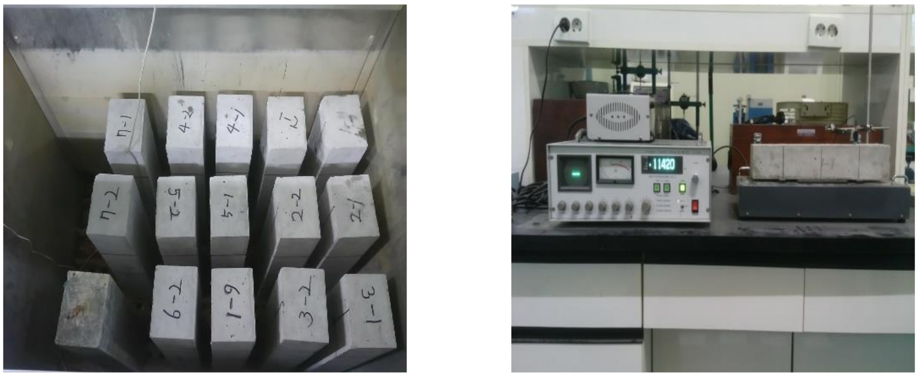
The columnar (Ø100 × 200 mm) concrete specimens were prepared according to KS F 2403 [39]. The compressive strength was measured according to KS F 2405 [40] after 3, 7, and 28 days of curing.

2.3.2. X-Ray Diffraction (XRD) Analysis

The phase analysis of hydration products was carried out by XRD (Bruker AX-S GmbH, Karlsruhe, Germany). The XRD was performed at 5°/min from 5° to 40°.

2.3.3. Freeze–Thaw Resistance of Concrete

Freeze–thaw resistance (Sanyo digital type Young's modulus rigidity meter, Japan) was tested according to ASTM C 666 [41]. The test specimen used in the experiment was a cuboid, i.e., 101 mm × 76 mm × 412 mm. One cycle of the freeze–thaw experiment was set to change from 4 °C to −18 °C for 2 to 4 h. The dynamic modulus of elasticity was measured repeatedly every 30 cycles. Figure 1a,b show the specimens placed in the freeze–thawing chamber and the equipment used to measure the freeze–thaw values, respectively.

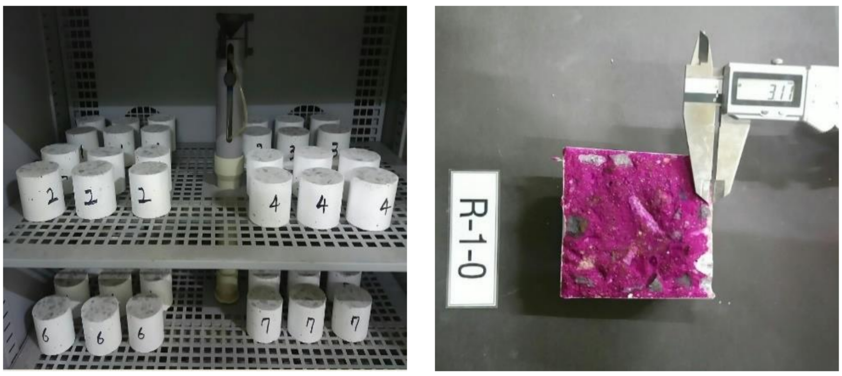


(**a**) (**b**)

Figure 1. (**a**) Freeze–thaw chamber; and (**b**) equipment.

2.3.4. Carbonation Resistance of the Concrete

The carbonation resistance of the concrete was performed on a cylindrical specimen (Ø100 × 200mm). The carbonation of concrete was performed in a carbonation chamber (HEUNGJIN, Incheon, South Korea) up to 91 days. The carbonation depth of the concrete specimens was measured according to RILEM CPC-18 [42] using a phenolphthalein solution. Figure 2a and b show the specimens kept in the carbonation chamber and carbonation depth measurement, respectively.

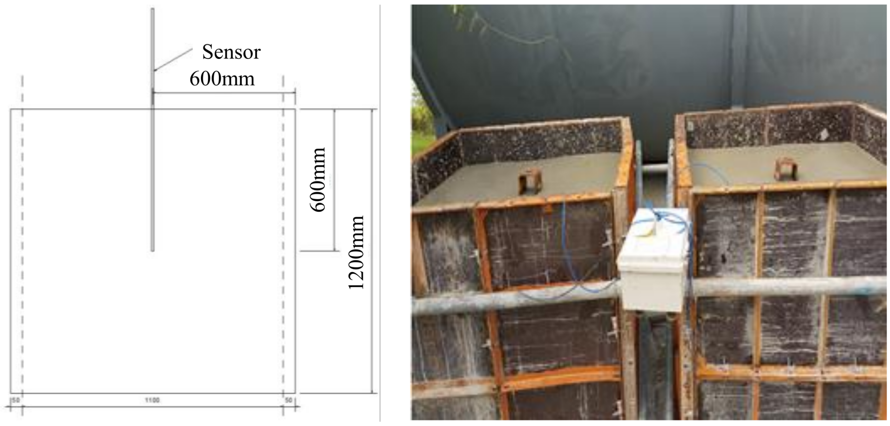


(**a**) (**b**)

Figure 2. (**a**) Concrete specimen kept in carbonation chamber; and (**b**) carbonation depth measurement.

2.3.5. Heat of Hydration

The heat of hydration was performed on 1200 mm × 1200 mm × 1200 mm concrete specimens. Prior to measuring the heat of hydration, a 100 mm thick polyurethane insulating material was used to avoid the temperature loss during measurement. A thermocouple was installed in the center of the mass concrete to measure the temperature, as shown in Figure 3a; the cast of concrete formwork is shown in Figure 3b.



(**a**) (**b**)

Figure 3. (**a**) Thermocouple embedded position; (**b**) the mass concrete in the formwork.

3. Results and Discussion

3.1. Compressive Strength

Figure 4 shows the compressive strength values of the concrete specimens. It can be seen from this Figure that the compressive strength of recycled water and activated sludge concrete specimens exhibited higher compressive strength compared to plain concrete. At 3 days of curing, the compressive strength of recycled water and activated sludge specimen was higher compared to plain concrete (Figure 4a), attributed to the higher pH of recycled water, which accelerated the hydration reaction of cement particles [43,44].

It can be seen from Figure 4b that as the curing duration was increased up to 7 days, the compressive strength of the specimens was around 24 MPa. Once the curing duration was increased up to 28 days, the compressive strength was increased appreciably and reached more than 33 MPa for all specimens (Figure 4c). In particular, it can be seen that the lower amount of cement used with activated sludge improved the compressive strength significantly compared to plain concrete.

3.2. X-Ray Diffraction Analysis

Figure 5 shows the XRD pattern of the plain and recycled water with desulfurized gypsum concrete (R-3-5). It can be seen from this figure that plain as well as R-3-5 concrete showed the C-S-H, C-A-H, ettringite, calcium hydroxide, and gypsum [43]. The ettringite and C-A-H led to the improved early strength of concrete, and thus the desired strength, i.e., 24 MPa, was observed by both types of concrete. Moreover, it can be seen in the R-3-5 specimen that the XRD intensity of the gypsum peak was most attributed to the desulfurized gypsum, which is the main component of activated sludge [43]. Gypsum from the activated sludge accelerated the hydration reaction of the cement; thus, ettringite and C-A-H were observed in XRD [44–47].

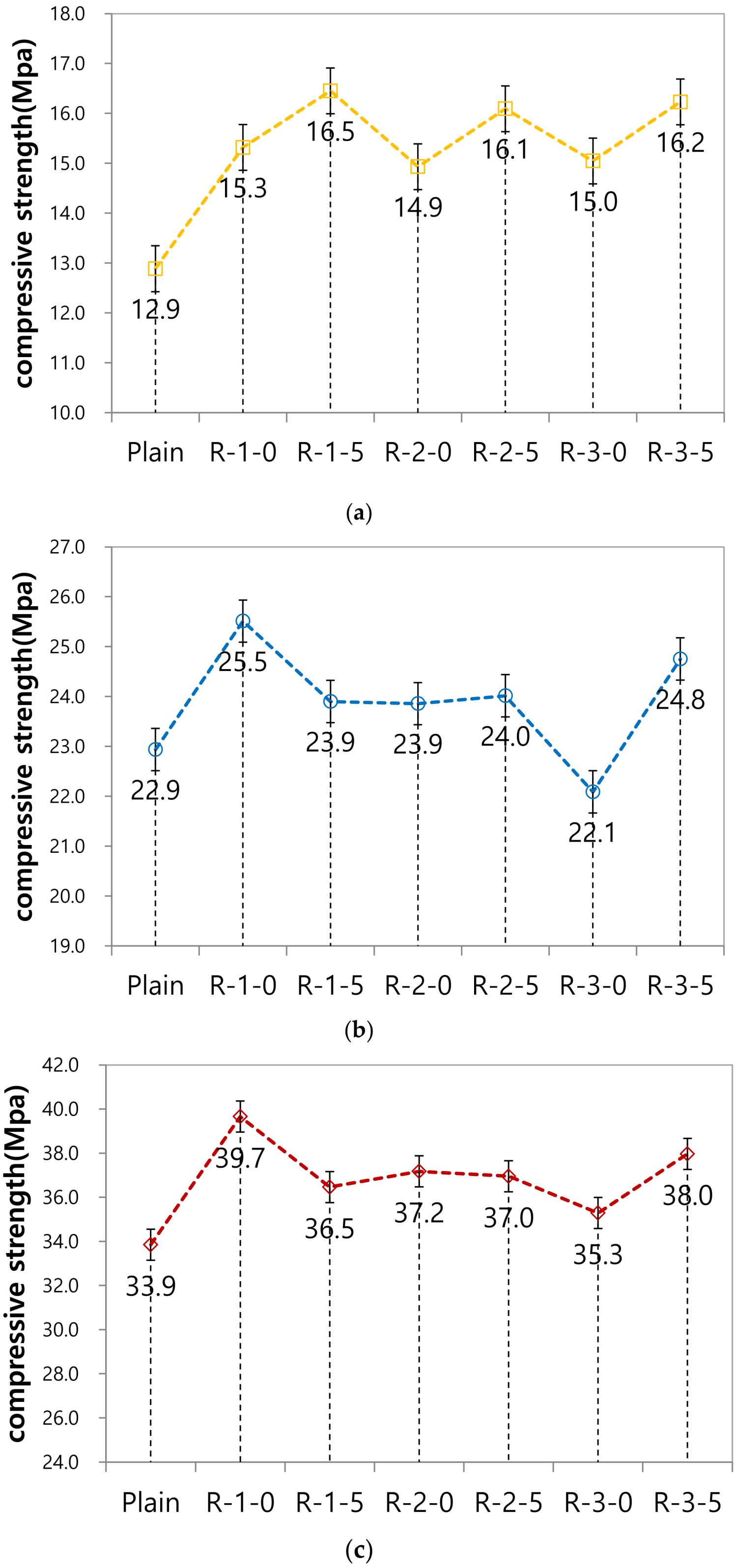


Figure 4. Compressive strength according to age: (**a**) 3 days, (**b**) 7 days, (**c**) 28 days.

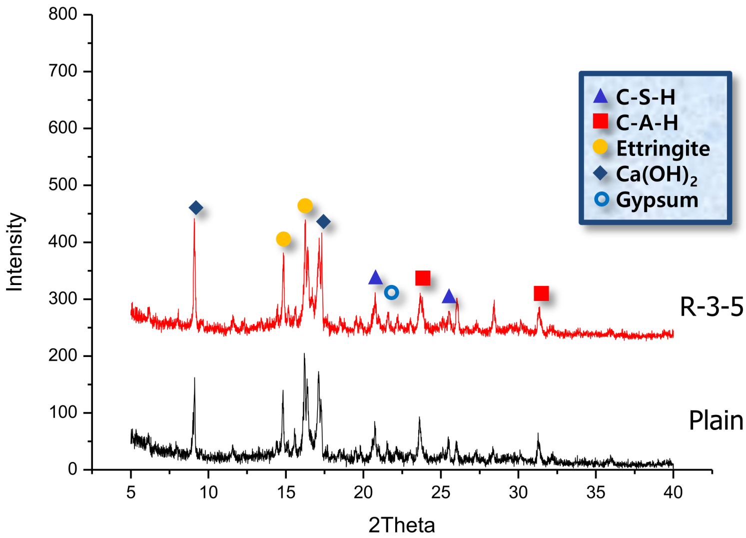


Figure 5. XRD pattern of concrete.

3.3. Evaluation of Freeze–Thaw Resistance

Figure 6 shows the calculation of the relative dynamic modulus elasticity using Equation (1) [41] after 300 cycles of freezing and thawing.

$$Pc = n_1{}^2/n^2 \tag{1}$$

where Pc = relative dynamic modulus of elasticity (%) after c cycles of freezing and thawing, n = fundamental transverse frequency at 0 cycles of freezing and thawing, and n_1 = fundamental transverse frequency after c cycles of freezing and thawing.

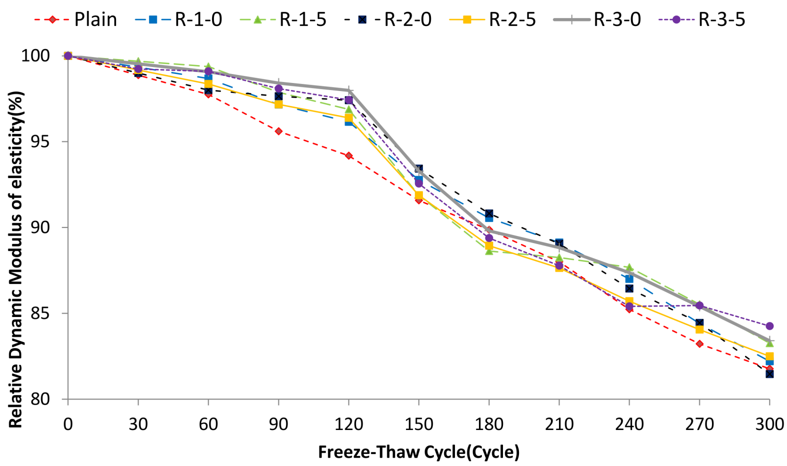


Figure 6. Relative dynamic modulus elasticity measured through freeze–thaw experiment.

The relative dynamic modulus elasticity of all specimens after 300 cycles was found to be more than 80% (Figure 6). It can be seen from Figure 6 that as the cycle was increased, the relative dynamic modulus elasticity was gradually decreased. From these results, it was found that the concrete mixed with 3% recycled water and 5% desulfurized gypsum, i.e., R-3-5, had higher resistance to freezing and thawing compared to plain concrete after 300 cycles.

Figure 7 shows the mass loss and reduction rate after 300 cycles of freezing and thawing. It can be seen that the mass reduction of the specimen was found to be plain > R-2-0 > R-1-0 > R-1-5 > R-3-0 > R-2-5 ≈ R-3-5. In terms of the mass reduction rate, the trend was almost identical, as observed in mass loss reduction. This result suggests that the greater amount of solid content in recycled water leads to a decrease in the mass loss as well as the rate of mass loss. In addition, when the desulfurized gypsum was used with the recycled water, the mass reduction was further reduced compared to recycled water. This result infers that the mass reduction and rate of other specimens was found lower than plain concrete, and it was in good agreement with the relative dynamic modulus. It might be attributed to the presence of $Ca(OH)_2$ and CaO in recycled water and desulfurized gypsum, which influences the formation of a dense structure and fills the pores of the concrete matrix.

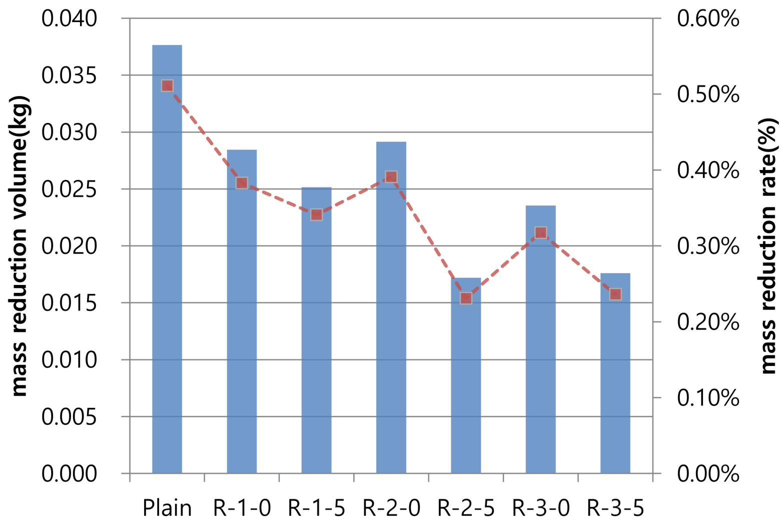


Figure 7. The mass reduction volume and rate.

The degree of durability resistance against freezing and thawing was evaluated by the durability factor (DF), as shown in Equation (2) [41].

$$\mathrm{DF} = \mathrm{PN}/\mathrm{M} \tag{2}$$

where P = relative dynamic modulus of elasticity at N cycles (%), N = number of cycles at which P reaches a specified minimum value for discontinuing the test or the specified number of cycles at which the exposure is to be terminated, whichever is less, and M = the specified number of cycles at which the exposure is to be terminated

Figure 8 shows the durability factor of each specimen. It can be seen from this Figure that the DF index of all specimens was greater than 80 and was within the satisfactory durability region. It is recommended that if the DF index is 100, then it is considered as durable concrete, but if this value is more than 60 and less than 100, then it is satisfactory. Moreover, if this value is between 40 and 60, then it can be said that the concrete is reliable. Sometimes, the concrete shows a DF value less than 40, and then it can be said that such concrete has a serious durability issue.

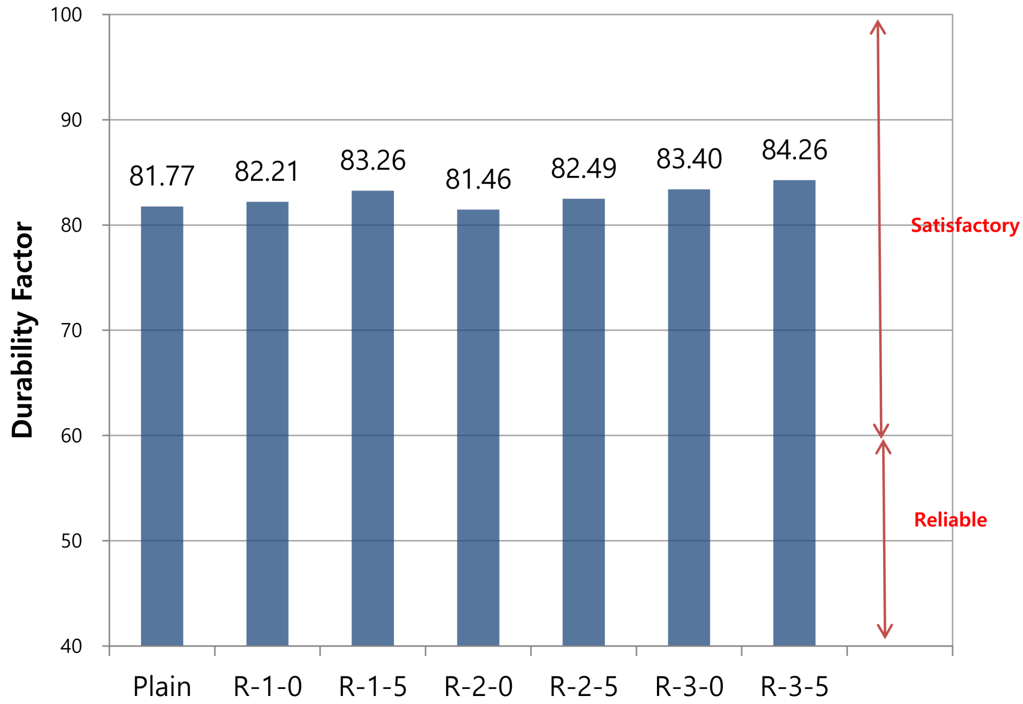


Figure 8. Durability factor.

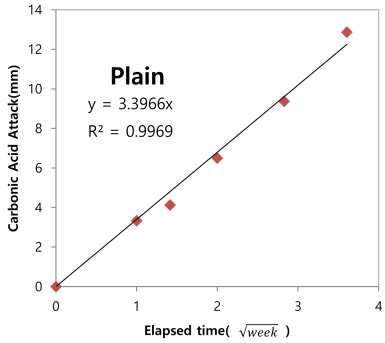
3.4. Evaluation of Carbonation Resistance

Figure 9 shows the carbonation depth of each specimen over time; the carbonation rate coefficient was derived through linear regression analysis. A typical carbonation model is shown in Equation (3), modified from Fick's first law.

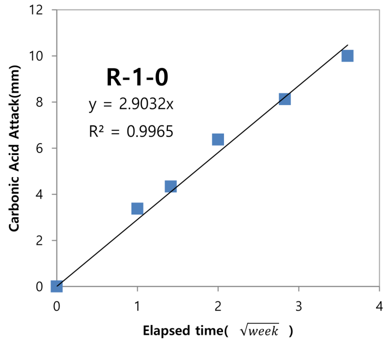
$$X_c = K \cdot \sqrt{t} \tag{3}$$

where X_c is the carbonation depth (mm), t is the carbonation period (year), and K is the carbonation rate coefficient ($mm/\sqrt{week}$).

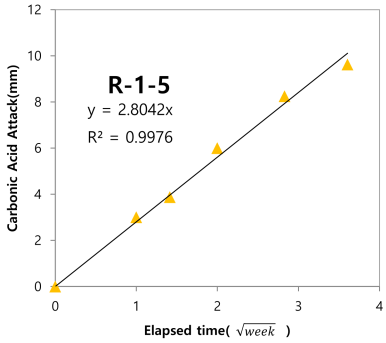
Carbonation depth of all specimens was measured at 1, 2, 4, 8, and 13 weeks of exposure, and it was found to be 3.00–3.38, 3.5 0–4.33, 6.00–6.63, 8.00–9.38, and 9.63–12.88 mm, as shown in Figure 9, respectively. The carbonation rate coefficient was calculated by considering the carbonation depth for each age of exposure and was found to be 2.833–3.397, and the correlation coefficient values for all specimens was 0.95 or higher, which indicates high reliability. Moreover, it can be seen that all specimens showed identical carbonation penetration depth until 8 weeks, but the highest carbonation penetration depth was observed with plain concrete, while the lowest was observed with the R-3-5 specimen after 8 weeks of exposure, which was attributed to the accelerated rate of hydration and pozzolanic reaction of blast furnace slag and fly ash, which make the concrete dense [43,44,48].



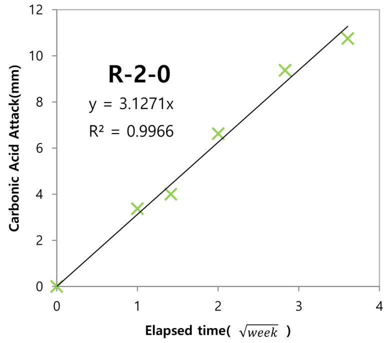
(**a**) Plain



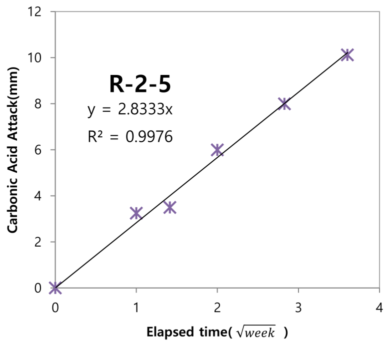
(**b**) R-1-0



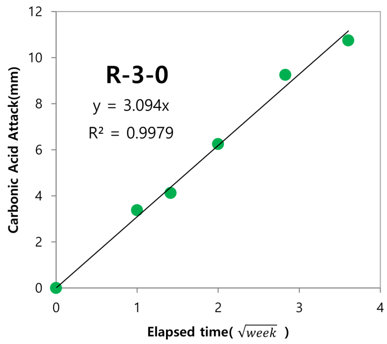
(**c**) R-1-5



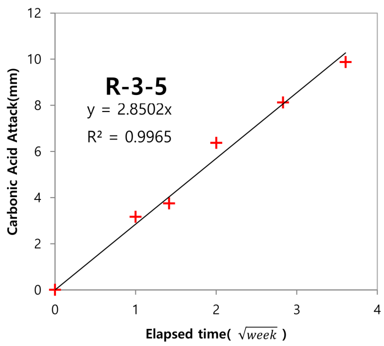
(**d**) R-2-0



(**e**) R-2-5



(**f**) R-3-0



(**g**) R-3-5

Figure 9. Carbonation depth with time.

3.5. Heat of Hydration Evaluation for the Concrete

A thermocouple was installed in the center of the test specimen to investigate the heat of hydration characteristics of plain concrete and activated sludge concrete (R-3-5), and the temperature was measured at 4 h intervals until it was stabilized. Figure 10 shows the core temperature history of plain concrete and activated sludge concrete. In the case of plain concrete, a maximum of 42.2 °C was attained after 60 h, but beyond this period, the temperature gradually decreased. However, in the case of activated sludge concrete (R-3-5), the maximum temperature was reached at 47.1 °C after 44 h. Moreover, until 20 h, the rise in temperature of plain and activated sludge concrete (R-3-5) was found to be identical, but once the elapsed period was increased, the rise in temperature of activated sludge concrete (R-3-5) increased compared to plain concrete. In addition, it was confirmed that activated sludge concrete (R-3-5) not only took less time to reach the maximum temperature but also had a higher temperature compared to plain concrete. The activated sludge concrete (R-3-5) not only promoted the hydration reaction but also increased the pH towards alkaline, which was attributed to the presence of blast furnace slag and fly ash [49,50].

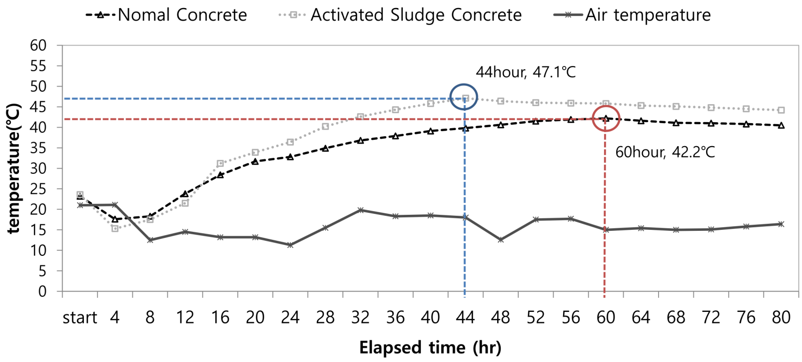


Figure 10. Plain concrete and activated sludge concrete temperature history.

4. Conclusions

In the present study, we evaluated the chemical properties, durability, and heat of hydration of concrete using activated sludge with desulfurized gypsum and recycled water. The XRD results of hydration products confirm the presence of C-S-H, C-A-H, ettringite, $Ca(OH)_2$, and gypsum. The highest intensity of ettringite and C-A-H reveal the early strength of concrete. The freeze–thaw resistance, carbonation resistance, and concrete heat of hydration confirm that using 5% activated sludge with recycled water improves the durability of the concrete. The presence of blast furnace slag and fly ash in activated sludge increases the pH of concrete, which accelerates the hydration and pozzolanic reactions. The accelerated hydration reaction causes the concrete temperature to rise. Therefore, it is suggested to use activated sludge (an industrial by-products) with recycled water to make durable concrete. From the present study, we can reduce the amount of cement and CO_2 and improve the environmental sustainability of concrete production using industrial by-products.

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