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Evaluation of the feasibility of using calcium aluminate composite (CAC) and Acement as additives for regulated set cement

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This work aims to evaluate the feasibility of using calcium aluminate composite (CAC) manufactured from steel slag, quickly cooled with high pressure air, and Acement, produced from CAC and various additives including gypsum as enhancements for regulated set cement. To evaluate the performance CAC or Acement as the additives of regulated set cements, CAC and Acement were replaced for the commercially available calcium sulfoaluminate cement (CSA)-based rapid setting cement (RSC). The engineering properties of workability, strength, and elastic modulus of mortar and concrete phases were then tested. From the mortar tests, as the replacement ratio of CAC was increased, the compressive strength of the mortar significantly decreased because of reduced amounts of gypsum for ettringite formation. On the other hand, as the replacement ratio of Acement including gypsum was increased, the compressive strength increased due to enhanced ettringite formation. From the test results, Acement displayed more favorable results as an additive for the rapid setting cement than CAC. CAC continued to show good performance at less than 10% of replacement ratio. Concrete testing revealed that mechanical performances of compressive, tensile, and flexural strength and elastic modulus all improved by 10% with CAC replacement and by 20% with Acement replacement. However, the resistivity performances for freeze-thaw tests and carbonation slightly decreased.

Key words: Ultra-rapid setting cement, Rapidly-cooled steel slag, CAC, Acement.

Introduction

In the cement and concrete industry, many researchers [1-3] advocate recycling various byproducts to improve industrial competitiveness and sustainability, thus developing "green technology." According to the statistics of the Korean Steel Association [4], the production of steel products is increasing; hence the amount of steel slag, a byproduct from the manufacturing process of steel products, is also increasing. Hence, the demand for research on the reutilization of steel manufacturing byproducts is increasing.

Steel slag is categorized as either converter slag or electric arc furnace slag. Additionally, electric arc furnace slag is categorized as either ladle furnace slag or oxidizing slag [5]. In South Korea, most steel slags are dumped in the cooling yard without any distribution or water-based aging treatment. Finally, these steel slags have only been used as low valued material, such as for filler aggregate.

One of the most critical reasons for limited recycling of steel slag is the nature of expansion caused by free lime (free-CaO; hereafter referred to as f-CaO). According to previous research [6], by utilizing rapid-cooling methods using high pressured air, it was possible to produce steel slag with a hard glassy surface, which can inhibit elution of f-CaO. Furthermore, it was reported that ladle furnace slag produced with this method contains Belite (β-C_2S) and Mayenite ($C_{12}A_7$)[1] so it was possible to apply it as an additive or supplementary material for cement-based materials [7, 8].

Therefore, in this research, the mortar and concrete were prepared with a mixture of commercially available $C\bar{S}A$ type rapid setting cement, and newly produced rapid setting cement (Acement) using CaO-Al_2O_3 composites (hereafter referred to as CAC) produced by quick cooling of molten ladle furnace slag by high-pressured air. Using the mortar phase, the optimum ratio of CAC or Acement is identified by fundamental tests of fresh state and hardened states. Based on the optimum ratio obtained from the mortar experiment, various engineering properties in the concrete phase were evaluated.

Theoretical Background

Occurrence of ladle furnace slag and stabilization

The electric arc furnace process has several functions: 1) Oxidation removes impurities such as Si, Mn, Cr, and P by the oxidizing reaction of the solvent.

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2) The ladle process controls the chemical composition of molten irons by stabilizing the temperature, introducing a deoxidizer and ferro alloy, controlling the shape of non-metallic inclusions, and removing impurities, such as P, S (SiO_2), C, (CaO), N, and H (H_2O or OH). The slags occurring in this ladle process represent approximately 15% of all electric arc furnace slag.

The f-CaO, normally existing in ladle furnace slag, results from the incomplete reaction of quicklime with other impurities during the refining process. Since the f-CaO in the ladle furnace slag is a major cause of poor volume stability, a stabilizing treatment is essential before using this slag as a construction material. There are some methods of aging, which stimulate reaction of f-CaO with moisture by long-term exposure outdoors, producing a hard glassy surface on slag. The most common methods of stabilizing steel slag are spraying high-pressured air or water on the molten material. This reaction produces f-CaO as a stable phase by adding additives and oxygen to the molten slag [9, 10].

Chemical compositions and constituent minerals of rapidly cooled ladle furnace slag

Because of the large amount of flux used to remove impurities of P, S, C, N, and H in molten iron during the ladle furnace process, ladle furnace slag has high concentrations of CaO, Al_2O_3, and SiO_2. By rapidly cooling this ladle furnace slag with high-pressure air, most slag turns amorphous as shown in Fig. 1 of the XRD analysis results. Since the amorphous compound in ladle furnace slag is to evaluate the compounds, the sample was re-calcined at 900 ºC and slowly cooled down for XRD analysis. From the compound of ladle furnace slag, $C_{12}A_7$, and β-C_2S were detected. On the other hand, the f-CaO is removed by immediate cooling in the reaction of CaO with Al_2O_3, and SiO_2 [11]. For crystalized compounds, $C_{12}A_7$ produces the hydration product of CAH_{10} at ordinary temperature [12], but thermodynamically converts it to C_3AH_6, a metastable hydration product through $C_3AH_{8.}$ [13], and this process of producing a metastable intermediate hydration product causes low strength [14]; hence, gypsum should be added to produce ettringite [15] and monosulfate as shown in Equation (1), thereby delaying the hydration speed and achieving stable and improved strength [16] of the mixture. Additionally, β-C_2S, the same compound found in Portland cement, dominates the long-term strength development [17].

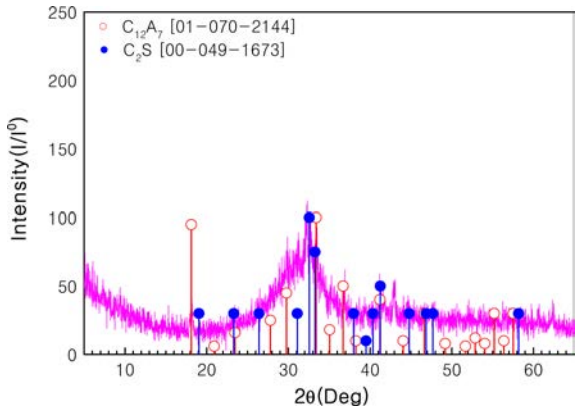

Fig. 1. XRD pattern of ladle furnace slag.

$$C_{12}A_7 + 12CaSO_4 + 137H_2O \rightarrow 4(C_3A \cdot 3CaSO_4 \cdot 32H_2O) + 6AH_3 \quad (1)$$

Experimental

Material properties

The mortar experimental plan for identifying optimum replacement ratio of CAC or Acement is shown in Table 1. Based on the control mixture with RSC from "S" company, the water-to-binder ratio was fixed at 0.40 with a cement-to-sand ratio of 1 to 3. The target mini slump was designated as 110 ± 12.5 mm. For the mortar experiment, the RSC from a single company (S company) was used. The replacement ratios of CAC or

Table 1. Experiment plan for mortar.

Mixtures	W/B*	0.40
	C : S**	1 : 3
	Binder (plain)	$C\bar{S}A$***-based regulated set cement (RSC from "S" company)
	Additive	CAC or Acement
	Replacement ratio (% by binder weight)	0, 10, 20, 30
Tests	Fresh mortar	Temperature Mini slump
	Hardened mortar	Compressive strength (3 h, 1, 3, 7, 14, 28D)

*W/B: water-to-binder ratio.
**C: cement, S: sand.
***$C\bar{S}A$: Calcium sulfoaluminate (one of the most representative types of regulated set cement).

Table 2. Mixing proportion of mortars.

ID*	C:S**	W/B***	Unit weight (kg/m^3)****						
			W	RSC	CAC	Ace	AD	Re	S
Plain	1 : 3	0.40	180	450	–	–	-	0.9	1350
C10				405	45	–	0.45	1.5	
C20				360	90	–	0.90	2.1	
C30				315	135	–	1.35	2.7	
A10				405	–	45	1.6	1.6	
A20				360	–	90	2.3	2.3	
A30				315	–	135	3.1	3.1	

*C10: CAC 10 wt. % replacement, A10: Acement 10 wt.% replacement.
**C:S: cement : sand.
***W/B: water-to-binder ratio.
****W: water, RSC: commercially available rapid setting cement, Ace: Acement, AD: admixture (superplasticizer), Re: gypsum as a retarder, S: sand.

Table 3. Experiment plan for concrete.

Mixture	Unit water (kg/m3)		160
	Unit cement (kg/m3)		400
	Plain binder		RSC* from "S" company RSC* from "U" company
	Additives replacement		CAC 10 wt. % Acement 20 wt. %
Tests	Fresh concrete		● Slump ● Air content ● Discharged temperature
	Hardened concrete	Mechanical properties	● Compressive strength (3H, 1, 3, 7, 14, and 28 D) ● Tensile strength (28 D) ● Flexural strength (28 D) ● Elastic modulus (28 D) ● Autogenous shrinkage
		Durability	● Freeze-thaw resistivity ● Carbonation depth

*RSC: rapid setting cement.

Acement were prepared at 0, 10, 20, and 30% for four different conditions. The temperature and mini-slump were measured for fresh state mortar. For hardened properties, compressive strength was measured every three hours on days 1, 3, 7, 14, and 28. The mixing proportions of mortars with various replacement ratios of CAC or Acement are summarized in Table 2.

The experimental plan for concrete incorporating CAC or Acement is summarized in Table 3, which shows two different rapid setting cements from different companies with a water-to-binder ratio fixed to 0.40. The mix design was conducted to satisfy a 210 ± 25 mm target slump and 4.5 ± 1.5% target air content. Based on this design, CAC 10% or Acement 20% by weight was substituted for rapid setting cement. Both CAC and Acement underwent the same tests at different composition percentages. Six batches were prepared from different companies (S & U): Plain cement mixes are identified as S-RSC & U-RSC; Acement mixes are SA20 and UA20; CAC mixes are S-C10 and U-C10. Fresh state property measurements were based on discharging temperatures recorded right after the mixing process as well as slump and air content. Hardened property measurements were based on compressive strength, tensile strength, flexural strength, elastic modulus, and autogenous deformation. The compressive strength of the concrete was measured at three hours and 1, 3, 7, 14, and 28 days. Special attention was given to evaluate early age compressive strength development at 3-hours, 1-day, and 3-day. For other mechanical properties, the tests were conducted in 28 days. Freeze-thaw and carbonation resistivity tests were used to evaluate durability performance. Table 4 shows the mixing proportions for each concrete mixture.

Sample preparation

In this research, rapid setting cements used were obtained from two different sources, that is, the "S" and "U" companies in South Korea. For the mortar properties experiment, RSC from "S" company was used. For the concrete properties experiment, RSC from both "S" and "U" companies were used and compared. For the RSC using CAC and Acement additives, CAC was produced by crushing the rapidly cooled ladle furnace slag, while Acement was produced by adding gypsum and other additives as setting time controllers in CAC. Table 5 shows the material components for manufacturing Acement. Each binder's physical properties provided by manufacturer and chemical compositions obtained by XRF are shown in Table 6.

The aggregate used in the mortar experiment was ISO standard sand obtained from South Korea. For the concrete experiment, the fine aggregate was natural river sand obtained from Kangwon-do, South Korea. Coarse aggregate was the 25 mm grade crushed

Table 4. Mixing proportions of concrete.

ID*	W/C**	S/a***	Unit weight**** (kg/m3)								
			W	RSC	CAC	Ace	S	G	AD	AE	Re
S-RSC	0.40	0.50	160	400	–	–	861	881	4	0.12	0.8
S-C10				360	40	–	862	881			
S-A20				320	–	80	861	880			
U-RSC				400	–	–	863	882			
U-C10				360	40	–	863	882			
U-A20				320	-	80	862	881			

*S-RSC: "S" company rapid setting cement, S-C10: "S" company rapid setting cement based CAC 10% replacement, U-A20: "A" company rapid setting cement based Acement 20% replacement.
**W/C: water-to-cement ratio.
***S/a: Sand-to-aggregate ratio.
****W: water, RSC: rapid setting cement, Ace: Acement, S: sand, G: gravel, AD: admixture (superplasticizer), AE: air entrainer, and Re: gypsum as a retarder.

Table 5. Physical and chemical properties of binders.

Binder*	L.O.I (%)	Density (g/cm^3)	Blaine (cm^2/g)	Chemical compositions (%)**					
				SiO_2	Al_2O_3	Fe_2O_3	CaO	MgO	SO_3
"S" RSC	2.3	2.91	5,854	11.00	11.30	2.66	49.90	1.70	12.10
"U" RSC	2.8	2.93	4,697	10.40	17.20	1.40	54.50	1.40	11.40
CAC	1.2	2.95	5,600	19.40	26.60	1.32	43.00	6.41	2.21
Acement	2.4	2.85	5,250	22.70	20.00	1.01	38.90	4.72	8.20

*"S" RSC: rapid setting cement from "S" company, "U" RSC: rapid setting cement from "U" company.
**obtained from XRF.

Table 6. Physical properties of aggregates.

Aggregate	Fineness modulus*	Density (g/cm^3)	Absorption rate (%)
Fine aggregate	2.98	2.62	2.11
25 mm coarse aggregate	7.13	2.68	1.37

*Fineness modulus was measured using the standard sieve set of ASTM C136 [18].

Table 7. Physical properties of chemical admixtures.

Type	Base	Phase	Color	Density (g/cm^3)	pH
Water reducer	Naphthalene sulfonic acid	Powder	Brown	1.5	–*
Retarder	Citric acid	Powder (Granular)	White	–*	2-3**

*not provided by manufacturer.
**measured in suspension phase.

aggregate from South Korea. The physical properties of the aggregates used in the concrete experiment are shown in Table 6 [18].

The water reducer used was a naphthalene-based powder type water reducer from "K" company in South Korea. To prevent rapid setting during the mixing process, a dose of retarder was added. The retarder used was a commercially available product in South Korea. The physical properties of chemical admixtures are shown in Table 7.

The mortar was mixed with a planetary mixer. The mixing protocol was (1) add cement, fine aggregate, and water at the same time and mix for 60 seconds at the first speed, then (2) mix for 60 seconds at the second speed. For concrete mixtures, a twin-shaft mixer was used. The mixing protocol was (1) mix dry mix binder and aggregate for 20 seconds at low speed (20 srpm), then (2) add water, retarder, and air entrainer at high speed mixing (40 rpm) for 180 seconds. The mixed specimens were demolded after one day and cured in a water bath at 20 ± 2 °C until removal at required age.

Tests methods

To evaluate the fresh state mortar properties, workability was measured using a mini-slump test following the KS F 2476 standard (similar to ASTM C143 [19] but with smaller cone dimension of 50 mm in the upper circle, a diameter of 100 mm in the lower circle, and a 150 mm height). The fresh state tests were conducted three times for repeatability, and the results were obtained by averaging the measured values. For hardened mortar properties, compressive strength was measured on a 50 mm cubic specimen using 3000 kN UTM following ASTM C109 [20] at given ages. Each measurement was performed three times, and the end results have averaged values.

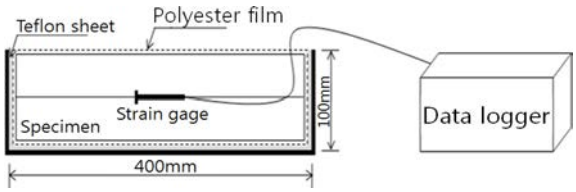

Fig. 2. Schematic test setting for autogenous shrinkage.

The fresh state properties of concrete, slump and air content were measured following ASTM C143 and C231 [21] methods, respectively. All fresh state tests were conducted with three different samples obtained from different areas in the mixed concrete. The mechanical properties of the hardened concrete were determined on specimens cast according to ASTM C39 [22] protocols. Compressive strength, split tensile strength, and elastic modulus were measured following ASTM C39, C496 [23], and C469 [24], respectively, at the scheduled designated curing times. For the flexural strength test, a specimen beam was cast with a length of 400 mm × 100 mm (height) × 100 mm (depth), and the test was conducted following ASTM C78 [25] according to its prefixed curing times. For autogenous shrinkage, the test method suggested by the Japan Concrete Institute [26] was utilized. The deformation of specimens was measured with an embedded gauge as shown in Fig. 2. The tests measuring the concrete's mechanical properties were conducted with three specimens, and the autogenous shrinkage test was performed with two specimens to assure accuracy of the results, which were averaged.

To evaluate the durability of the concrete with various additives, freeze-thaw tests and carbonation resistances were measured. Freeze-thaw tests were conducted using another 400 mm × 100 mm × 100 mm

beam specimen following ASTM C666 [27] after 14 days of curing. The accelerated carbonation test [28] was performed on the same size specimens, which were cured in a water bath for 28 days and stored at a 20 ± 2 ℃ temperature, and 60 ± 5% relative humidity for 56 days in a CO_2 chamber with 5% CO_2 concentration. After preparation, the specimens were stored in the CO_2 chamber until testing at 1, 4, 8, and 13 weeks. To test, the specimens were cut perpendicular to the longitudinal direction of the beam and a 1% phenolphthalein suspension was applied on the cross section of the specimens at 60 mm from the edge. The carbonation depth was obtained as the average of 10 points on two surfaces.

Results and Discussion

Mortar properties

Figs. 3 and 4 show the properties of fresh state mortar depending on different replacement ratios of rapid setting additives. As shown in Fig. 3, all mixtures with rapid setting additives showed higher discharging temperature than plain RSC, and the discharging temperature increased with the increasing replacement ratio of rapid setting additives. It was determined that

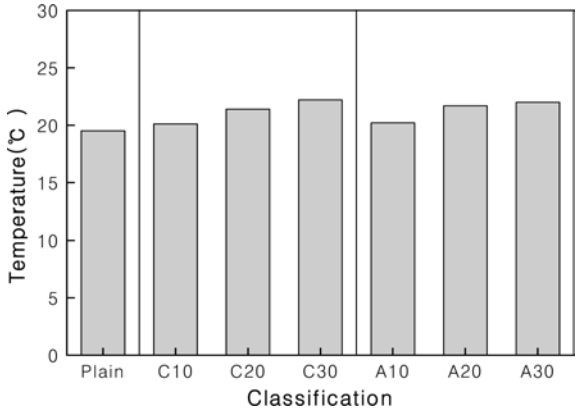

Fig. 3. Effect of replacement of rapid setting additives on temperature after mixing process.

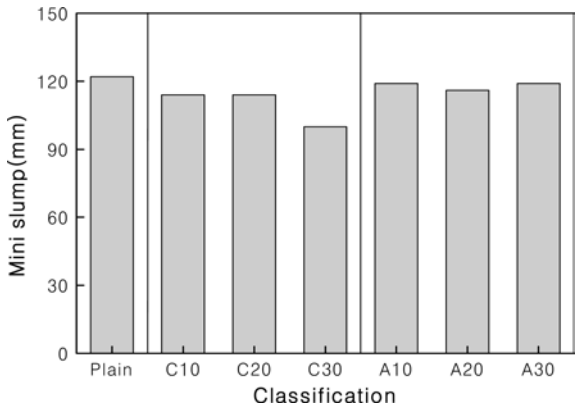

Fig. 4. Effect of replacement of rapid setting additives on mini-slump.

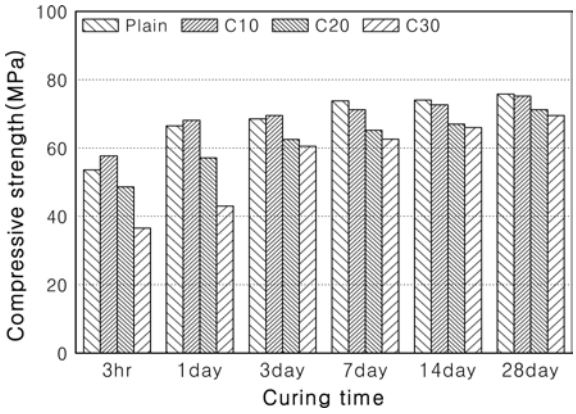

Fig. 5. Effect of replacement ratio of CAC on compressive strength depending on curing time.

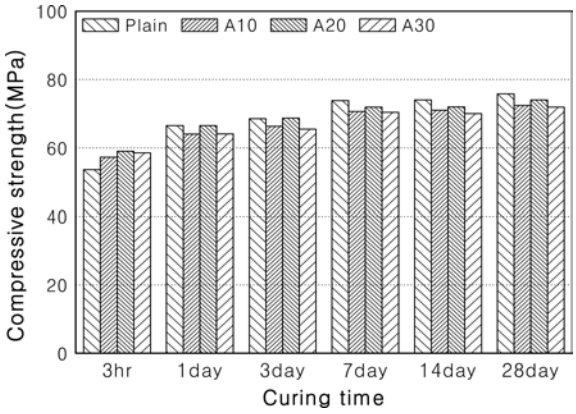

Fig. 6. Effect of replacement ratio of Acement on compressive strength depending on curing time.

the CAC and Acement with a main component of $C_{12}A_7$ have higher reactivity than plain rapid setting cement (RSC). The mini-slump of all mixtures satisfied the target slump range (see Fig. 4). From this result, it can be stated that the fresh state mortar with rapid setting additives possesses sufficient workability while retaining fast reactivity.

The effect of replacement ratios of CAC on compressive strength of the mortars depending on ages is shown in Fig. 5. Generally, with a CAC replacement ratio of 10%, higher compressive strength values were shown at the end of the three-day curing process. After three days of curing, compressive strength values comparable to the plain mixture were obtained. $C_{12}A_7$ is a main component of CAC and produces ettringite as a result of the reaction with $CaSO_4$ (see Equation (2)) This fast formation of ettringite is considered the reason for higher compressive strength at early age. (before three days of curing is completed), which is higher than that of the RSC plain mixture. On the other hand, when the mixture contains over 20% CAC, compressive strength values were lower than that of the plain mixture regardless of age. It can be stated that the amount of $C_{12}A_7$, a main component of CAC, is not enough to consume all gypsum for ettringite, so C_3AH_6

of the low-strength trisoctahedral stable phase was produced after forming sub-stable phases of C_2AH_8, C_4AH_{19}, and C_4AH_{13}.

The effect of the replacement ratio of Acement on compressive strength of mortar depending on age is shown in Fig. 6. In this case, all the mixtures replacing Acement showed higher compressive strength than the plain mixture after only three hours. It is thought that, similar to the CAC results, ettringite formation contributes to the fast strength development within the three-hour curing process. Since after three hours, all mixtures showed a compressive strength lower than the plain mixtures, it can be considered that the amount of hydration product with C_3AH_6 is less than that of the CAC mixtures because of the lesser amount of CAC in Acement.

Consequently, based on the results of these experiments, it is possible to use CAC and Acement as rapid setting additives for RSC, with the optimum replacement ratios being 10% for CAC and 20% for Acement to RSC weight.

Concrete properties

Figs. 7, 8, and 9 show the fresh state properties of concrete depending on different binder conditions. In Fig. 7, all concrete mixtures with rapid setting additives show higher post-mix temperatures than Plain RSC mixtures, regardless of the RSC brands. CAC and Acement showed higher reactivity and better hydration than Plain RSCs. Moreover, the workability of the concrete mixtures satisfied the target range with the appropriate mix design (see Fig. 8). With 10% CAC content and 20% Acement content, workability was decreased regardless of the types of RSCs. This was due to slump loss caused by the fast reaction of CAC and Acement in RSC binders. Regarding air content of rapid setting concrete, all mixtures showed favorable results within the target range (see Fig. 9).

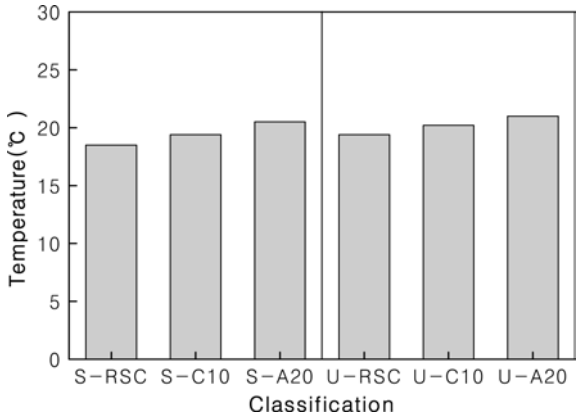

Fig. 7. Effect of various binders on temperature after the mixing process of the fresh state concrete.

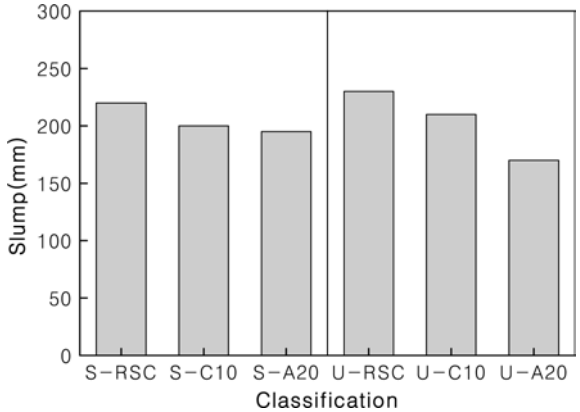

Fig. 8. Effect of various binders on slump of the fresh state concrete.

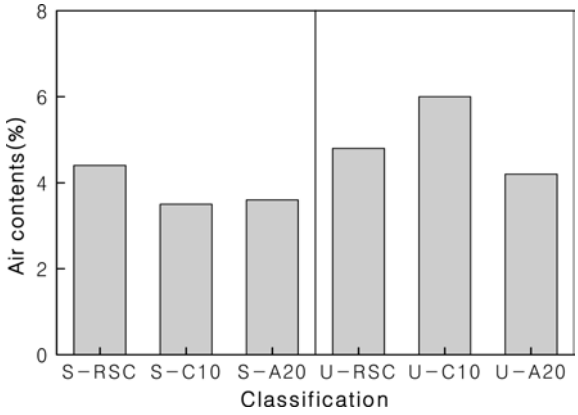

Fig. 9. Effect of various binders on air content of the fresh state concrete.

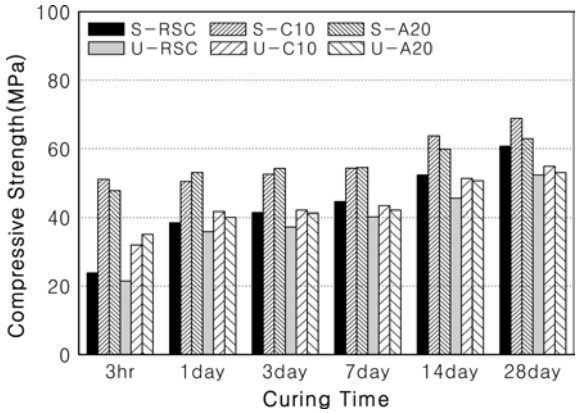

Fig. 10. Effect of various binders on compressive strength of the concrete mixtures.

(1) Compressive strength: Fig. 10 shows compressive strength test results depending on various binder conditions. Generally, the compressive strength of the mixtures with 10% CAC and 20% Acement was higher than that of the plain mixtures from “S” and “U” companies. This is attributed to the increased reactivity due to replacing CAC and Acement as well as mortar experiment results.

(2) Tensile strength, flexural strength, and elastic modulus: The mechanical performance test results for tensile strength, flexural strength, and elastic modulus are shown in Figs. 11-13, respectively. For all three

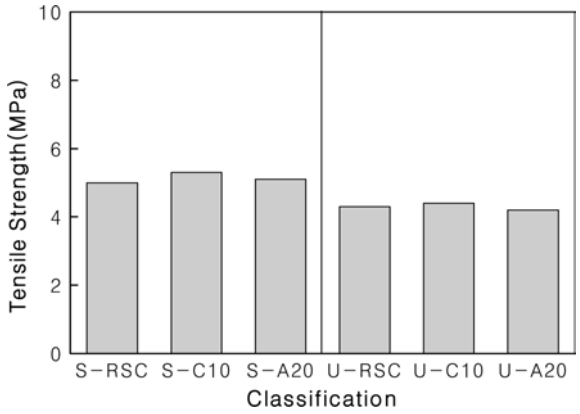

Fig. 11. Effect of various binders on tensile strength of different concrete mixture classifications.

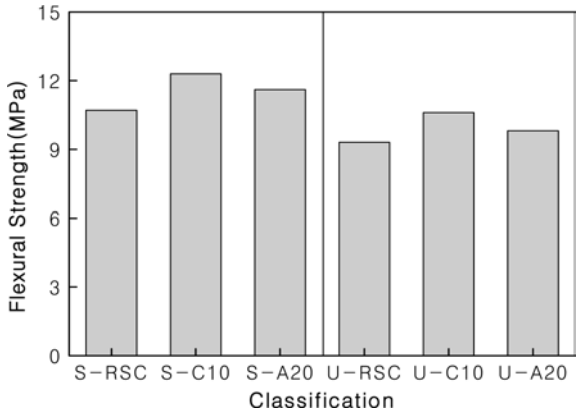

Fig. 12. Effect of various binders on flexural strength of different concrete mixture classifications.

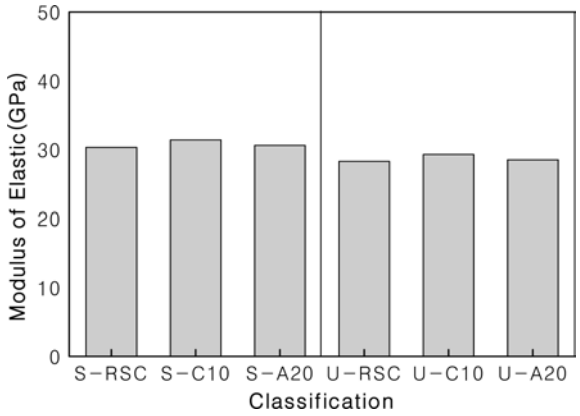

Fig. 13. Effect of various binders on elastic modulus of different concrete mixture classifications.

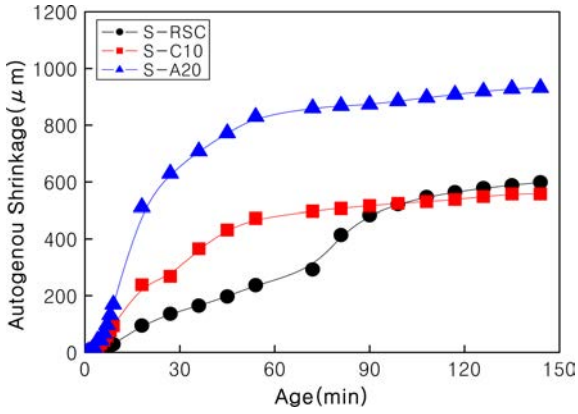

Fig. 14. Effect of various binders based on "S" RSC on autogenous shrinkage (within 150 minutes).

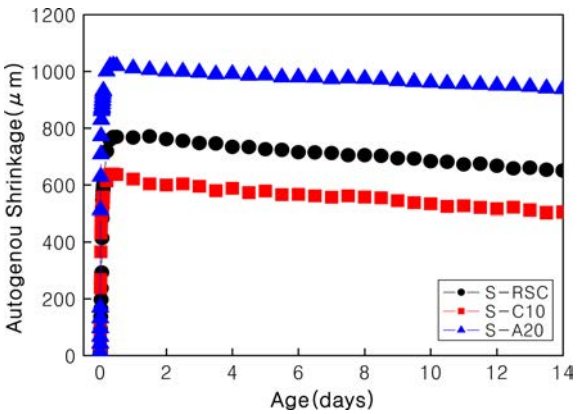

Fig. 15. Effect of various binders based on "S" RSC on autogenous shrinkage (within 14 days).

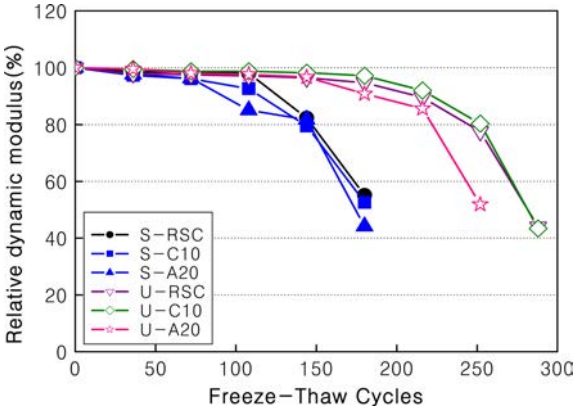

Fig. 16. Effect of various binders on relative dynamic modulus after freeze-thaw cycles.

mechanical tests, the concrete mixtures with CAC and Acement showed slightly improved performance. This can be related to compressive strength results.

(3) Autogenous shrinkage deformation: Figs. 14-15 show deformation of concrete specimens within 150 minutes (2.5 hrs) and within 14 days, respectively. Based on the figures, the mixture with 10% CAC showed high expansion at an early age, which means the expansion was similar to plain mixture after 90 minutes (see Fig. 15). This is considered a result of the fast formation of ettringite by the reaction of CAC following transformation to monosulfate because of insufficient gypsum for further ettringite formation. However, in the case of the mixture with 20% Acement, the expansion was higher than that shown in other mixtures and this expansion continued to the end of the testing period because of the additional gypsum from the process of Acement production from CAC.

(4) Freeze-thaw resistivity: Figs. 16-17 show the relative dynamic modulus and weight change, respectively, after

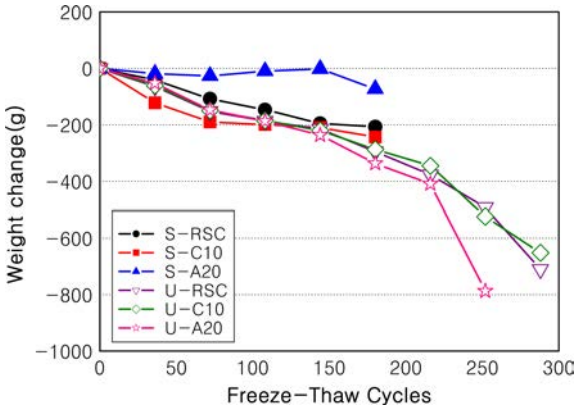

Fig. 17. Effect of various binders on weight change after freeze-thaw cycles.

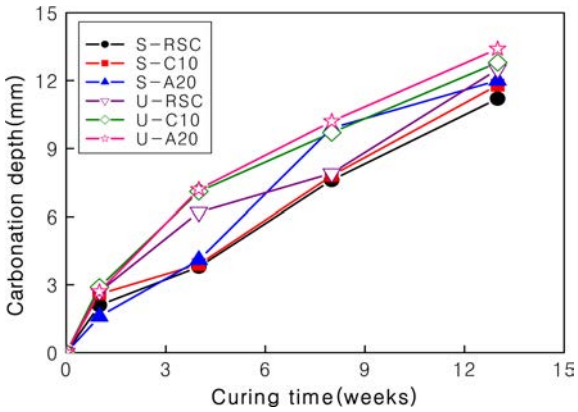

Fig. 18. Effect of various binders on carbonation depth depending on the curing time.

the freeze-thawing test. Among the two plain commercial RSC products, "U" company's RSC showed less degradation of relative dynamic modulus and weight change than "S" company's RSC. Furthermore, as rapid setting additives of CAC or Acement were replaced, the degradation of the relative dynamic modulus and weight change increased regardless of the RSC brands.

Accelerated carbonation depth: After the accelerated carbonation test, the carbonation depth of the specimens was graphed in Fig. 18. In respect to carbonation depth, the RSC from "U" company showed faster carbonation (deeper carbonation depth) than the RSC from "S" company. Replacing CAC or Acement in RSC increased carbonation depth regardless of the RSC brands.

Conclusions

This research analyzed the properties and compressive strength of CAC obtained from rapid air cooled molten steel slag and Acement manufactured from crushed CAC with gypsum and other additives. The results from these tests were compared to tests conducted on two types of plain cement from two different companies. Properties of mortar and concrete prepared with CAC or Acement binders based on a commercially available C$\bar{S}$A-based rapid setting cement were measured. Test results led to the following conclusions:

The results of the experiment for mortar properties: Experiments conducted to determine the replacement ratios of CAC and Acement on properties of mortar showed that as the replacement of CAC increased, mortar compressive strength decreased because of a lack of gypsum to produce sufficient amounts of ettringite. However, Acement with added gypsum in a CAC powder additive increased compressive strength. Gypsum was the key factor contributing to the compressive strength increase between CAC and Acement. Based on the mortar experiment-Acement, as the rapid setting additive for RSC, showed better performance than CAC within 10% of replacement ratio. The RSC mixture that replaced CAC also showed acceptable performance.

The results of experiment for concrete properties. When 10% CAC or 20% Acement content replaced RSC, the mechanical properties of compressive strength, tensile strength, flexural strength, and elastic modulus were improved. In spite of these improved mechanical properties, rapid setting additives of CAC and Acement decreased durability in freeze-thaw and carbonation resistivity tests. Therefore, further research should be performed for improving durability of RSC with CAC or Acement.

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