

Estimation of Setting Time of Cement Mortar combined with Recycled Aggregate Powder and Cement Kiln Dust based on Equivalent Age

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Abstract

This paper presents a method of estimating the setting time of cement mortar incorporating recycled aggregate powder (RP) and cement kiln dust (CKD) at various curing temperatures by applying an equivalent age method. To estimate setting time, the equivalent age using apparent activation energy (E_a) was applied. Increasing RP and CKD leads to a shortened initial and final set. E_a at the initial set and final set obtained by Arrhenius function showed differences in response to mixture type. These were estimated to be from 10~19 KJ/mol in all mixtures, which is smaller than those of conventional mixture ranging from 30~50 KJ/mol. Based on the application of E_a to Freisleben Hansen and Pederson's equivalent age function, equivalent age is nearly constant, regardless of curing temperature and RP contents. This implies that the concept of maturity is applicable in estimating the setting time of concrete containing RP and CKD. A high correlation was observed between estimated setting time and measured setting time. A multi regression model was provided to determine setting time reflecting RP and CKD. Thus, the setting time estimation method studied herein can be applicable to concrete incorporating RP and CKD in the construction field.

Keywords : recycled aggregate powder, cement kiln dust, equivalent age, setting time, curing temperature

1. Introduction

As the number of the urban re-development projects has been on the rise and old building structures have been made obsolete, the amount of waste concrete has been sharply increased. In the recycling of resources, waste concrete is now used as a recycled aggregate material. In the process of producing recycled aggregate, about 40 percent of the recycled aggregate powder (RP) is scattered or left unused[1,2].

Since RP is usually composed of CaO and SiO_2 elements similar to cement, and some is used as a

landfill material or a cover material after combining it with soil, or is left unused or scatters, it triggers secondary environmental pollution. When disposed as waste, high disposal costs are involved[3]. In other countries, including Japan, research has been actively carried out in the utilization of RP for materials for workability improvement and concrete aggregate, while in Korea few studies have been conducted on the effective recycling of RP.

For instance, Kim et al.[1] reported the results of an assessment of the usability of cement panel as alternative material to silica sand. Hyeon[4] suggested a method of manufacturing artificial lightweight aggregate by using the residue soil of construction. As well, Kang et al.[3] performed laboratory experiments on the properties by refiring in RP manufactured through a wet separation method, and assessed its usability. However, as of yet there has been no research

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conducted on recycling as aggregate to secure concrete setting and high early strength gain.

Thus, the researchers are studying the plan to utilize RP as early high strength aggregate for concrete by grading RP below 0.08 mm[5]. It is revealed that when RP was used as a portion of aggregate, the RP filled the pore space, and thus the strength of cement was increased. Here, when the cement kiln dust (hereinafter referred to as CKD), the powder generated in the process of cement manufacturing, is added, it is believed that the setting time can be reduced and the early high strength gain may also be improved, since CKD has a high amount of CaO, which facilitates hydration. In particular, according to Han et al.[6,7], CKD is reported to facilitate curing and improve strength under low-temperature conditions due to its high hydration activity and its high fineness, which exceeds 8,000 cm^2/g .

On the other hand, considering construction sites in Korea, there are changes in temperature with the season, and the curing and strength of concrete is affected by such temperature change. For this reason, it is important to have an accurate understanding of the effect of the setting temperature on concrete placement and maintenance. However, there has been no quantitative analysis of the temperature dependence of the setting reaction of concrete mortar mixed with RP and CKD, so research should be done in this area to enable the effective utilization of recycled resources in the future.

Therefore, the RR below 0.08 mm in the process of manufacturing recycled aggregate was collected and substituted as a fine aggregate. To secure an additional early high strength gain, CKD generated in the process of manufacturing cement was also substituted to conduct a quantitative analysis on the effect of changes in the temperature on the

setting reaction of the cement mortar combined with RP and CKD, and to estimate the setting time by applying an equivalent age method.

2. Design of experiment and method

2.1 Experimental plan

Table 1 indicates the experimental plan, and Table 2 shows the mixture proportions of mortar. First of all, as experimental factors, W/B was fixed at 50%, the mixture ratio of cement and aggregate at 1:3, and the amount of SP was determined to satisfy the target flow for the plain mixture of 150 ± 10 mm. The same amount of SP was used for the other concrete mortars. RP and CKD were substituted by mass based on the preceding study[5]. RP was substituted for fine aggregate in the proportion of 5%, 10% and 15%, respectively, while CKD was substituted for RP in the proportion of 10%, 20% and 30%, respectively.

Table 1. Experimental plan

Items		Factors	
Mixtures	W/B(%)	1	50
	C : S	1	1 : 3
	Target flow (mm)	1	150 ± 10 (Plain)
	RP (%)	10	0(Plain), 5, 10, 15
	CKD1)(%)		0(Plain), 10, 20, 30
	Curing temperature($^{\circ}\text{C}$)	3	● 5, 20, 35
Test	Fresh mortar	3	● Flow
			● Air content
			● Setting time

1) substituted to RP by mass

The curing temperature to estimate the setting was set at 5 $^{\circ}\text{C}$, 20 $^{\circ}\text{C}$ and 30 $^{\circ}\text{C}$, taking into account normal Korean temperatures in winter and summer. Flow, air content, and setting time were to be measured.

Table 2. Mixture proportions of mortars

Mixtures	Unit weight (kg/m ³)					
	Water	Cement	Sand	RP	CKD	Superplasticizer
Plain	172	383	1160	0	0	2
RP (5%)	CKD 10%	176	391	1125	53	6
	CKD 20%	175	390	1122	47	12
	CKD 30%	175	389	1119	41	18
RP (10%)	CKD 10%	180	399	1088	109	12
	CKD 20%	179	397	1082	96	24
	CKD 30%	178	395	1077	84	36
RP (15%)	CKD 10%	183	408	1050	167	19
	CKD 20%	182	404	1041	147	37
	CKD 30%	180	401	1033	128	55

2.2 Materials

The cement used in this study is ordinary Portland cement manufactured by Company A in Korea. The properties of the cement are indicated in Table 3. Crushed sand and river sand from Jochiwon, Chuncheongnamdo were used as aggregate by combining them in a 1:1 ratio. Table 4 shows the properties of each of the sands.

RP was collected from the production line of Company A using the dry separation method. RP below 0.08 mm was collected, and the properties of RP are indicated in Table 5. In Table 5, the properties of CKD, fine powder collected from the pre-heater section of Cement Company B, are also shown.

Polycarboxylic acid superplasticizer manufactured in Company B was used, whose properties are shown in Table 6.

Table 3. Physical properties of cement

Density(g/cm ³)	Blaine (cm ² /g)	Soundness (%)	Setting time (min.)		Compressive strength(MPa)		
			Initial	Final	3 days	7 days	28 days
3.15	3 165	0.07	232	429	24.5	33.1	43.9

Table 4. Physical properties of fine aggregates

Type	Density (g/cm ³)	Fineness modulus	Absorption ratio (%)	Unit weight (kg/m ³)	Passing amount of 0.08 mm sieve(%)
River sand	2.5	2.86	0.46	1518	0.30
Crushed sand	2.58	2.90	0.46	1684	0.32

Table 5. Physical and chemical composition of RP and CKD

admixture	Density (g/cm ³)	Blaine (cm ² /g)	L.O.I (%)	Chemical composition			
				SiO ₂	Fe ₂ O ₃	Al ₂ O ₃	CaO
RP	2.30	6 443	20.26	27.35	6.66	5.99	33.87
CKD	2.55	8 200	–	9.65	3.70	1.54	43.60

Table 6. Physical and chemical properties of high range water reducing agent

Type	Basis	Appearance	Density (g/cm ³)
High range water reducing agent	Polycarboxylate	Liquid, Dark brown	1.05

2.3 Experiment method

Mortar was mixed according to KS L 5109, and the flow of fresh mortar, air content, and the setting time were measured according to KS L 5105, KS F 2421 and KS F 2436, respectively. The setting time was measured in a chamber whose temperature was kept at 5°C~35°C after the mould was set.

3. Results and discussion

3.1 Flow of fresh mortar and air content

Figure 1 shows the flow values according to substitution by combining RP and CKD. First, the plain mixture satisfied the target flow, but as indicated in Figure 1, the higher the RP was, the lower the flow became. This seems to be because the substitution rate was increased as more RP was replaced for fine aggregate, and the viscosity accordingly became thicker. However, there were no significant differences in the flow values according to the substitution of CKD, which implies that although CKD was substituted for some of RP, it did not affect the overall powder content.

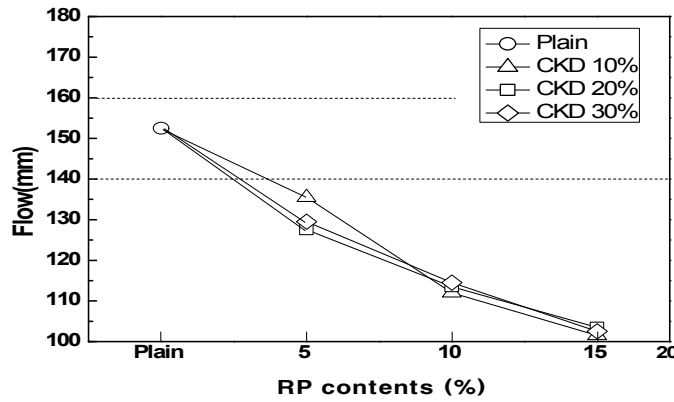


Figure 1. Flow with the contents of RP

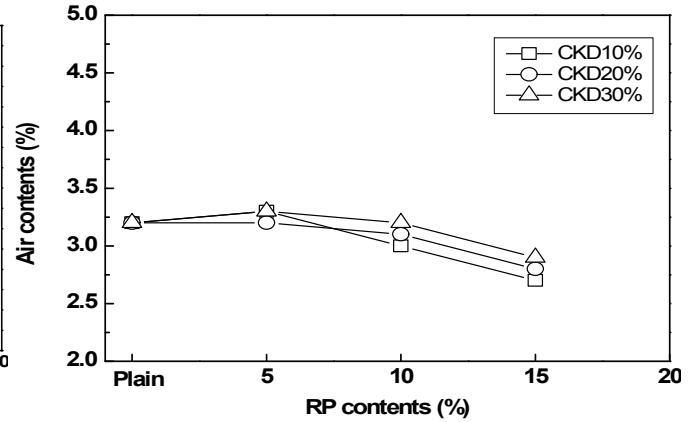


Figure 2. Air content with the contents of RP

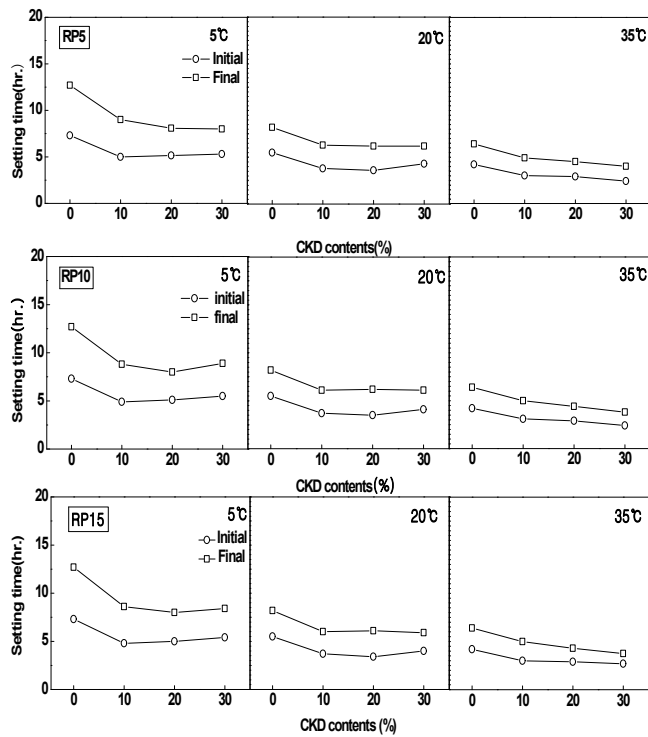


Figure 3. Setting time with the contents of RP and CKD

Figure 2 indicates air content according to changes in the substitution rate of RP and CKD. Overall, the more RP was substituted, the less the estimated air content, due to the effect of filling pore space. However, when more CKD was substituted, a slight decrease in air content was observed, but the overall decrease was within the error range, and the target air content was met.

3.2 Properties of setting according to curing temperature

Figure 3 shows the setting time by curing temperature according to changes in the substitution rate of RP and CKD. Overall, the higher the curing temperature, the more shortened the initial and final time was found to be, which means that hydration reaction is affected by the temperature. In other words, the higher the temperature becomes, the sooner the hydration reaction takes place. On one hand, there were no significant differences found according to the substitution rate of RP. This implies that although RP contains a lot of CaO, it does not facilitate hydration activity, and there is no significant change in the amount of cement mortar depending on the substitution of aggregate. On the other hand, the more the CKD was substituted, the sooner the cement was cured. When the proportion was up to 10%, the setting time was rapidly reduced compared to that of plain mixture, and was gradually reduced thereafter. It is believed that unlike that in RP, CaO in CKD facilitated the setting[4]. This was obvious at the curing temperature of 5°C, which confirmed the research finding of Han et al. that CKD facilitated the setting under a low-temperature condition.

3.3 Estimation of setting time

3.3.1 Procedure for estimating setting time

To conduct a quantitative analysis on the effect of changes in curing temperature on the setting reaction of cement mortar, the estimation method using equivalent age is discussed in this section.

For an interpretation of setting time, apparent activation energy (E_a) should first be calculated. In ASTM C 1074[7], the apparent activation energy was obtained by using Eq. (1). Setting and compressive strength of the cement mortar set at three different temperatures were measured by each age, a regression analysis was carried out with reciprocal strength and reciprocal age (at 2, 4, 8, 16, 32, and 64 times the setting time) to get the first regression equation. Then, the intercept of the regression line was divided by the slope to obtain the reaction rate constant, k_T .

$$k_T = A \cdot \exp\left(-\frac{E_a}{RT}\right) \quad \text{----- (1)}$$

$$\ln k_T = \ln A - \frac{E_a}{R} \cdot \frac{1}{T} \quad \text{----- (2)}$$

E_a can be calculated using Eq. (2). Obtained by a linear regression with the values of $\ln(k_T)$ and $1/T$ from experiment data, the slope of the first regression equation is E_a/R , and from this value, E_a can be obtained.

However, the Freisleben-Hansen equivalent age equation to be applied in this research is expressed as Eq. (3) using Arrhenius function. The equivalent age means the setting time at the standard temperature (20°C) where accumulated temperature becomes identical in an actual concrete setting.

$$t_e = \int_0^t \exp\left(\frac{E_a}{R}\left(\frac{1}{T_r} - \frac{1}{T}\right)\right) dt \quad \text{----- (3)}$$

Where,

E_a : apparent activation energy(KJ/mol)

R : gas constant(8,341 J/mol·K)

t_e : equivalent age

T_r : absolute temperature at 20°C(293 ° K)

T : curing temperature($t+273$ ° K)

To estimate the setting time based on equivalent age, a couple of processes and hypotheses are required. First of all, E_a at initial and final set should be obtained. At this time, it is assumed that fine structure is developed by the hydration reaction of concrete to some degree, even at initial and final set[9]. That is, if the degree of hydration is expressed as a_i and a_f at initial and final set, respectively, the following are the hypotheses that can be derived.

- 1) The time spent in the initial set is the same as the time taken to reach a_i .
- 2) The time spent in the final set is the same as the time taken to reach a_f .

Here, the sooner the hydration reaction takes place, the shorter the initial and final time is. That is why the time taken to reach initial and final set is in inverse proportion to the hydration reaction constant ($k_T \propto 1/t_i$). Under the condition and hypothesis above, E_a at initial and final set can be calculated using Arrhenius. Using the method proposed in ASTM C 1074, $\ln(1/t_i)$ is replaced with $\ln(1/t_i)$, the function relation with the reciprocal of the absolute temperature (T) is obtained, and a regression analysis is conducted on it to obtain E_a . The E_a obtained in this manner is substituted in Freisleben-Hansen equivalent age function to get E_a at initial and final set[10].

3.3.2 Estimation results of setting time

Figure 4 and Table 7 present the results of regression analysis of Arrhenius plot obtained using Eq. (2). Overall, the reciprocals of the measured setting time are shown to be proportional

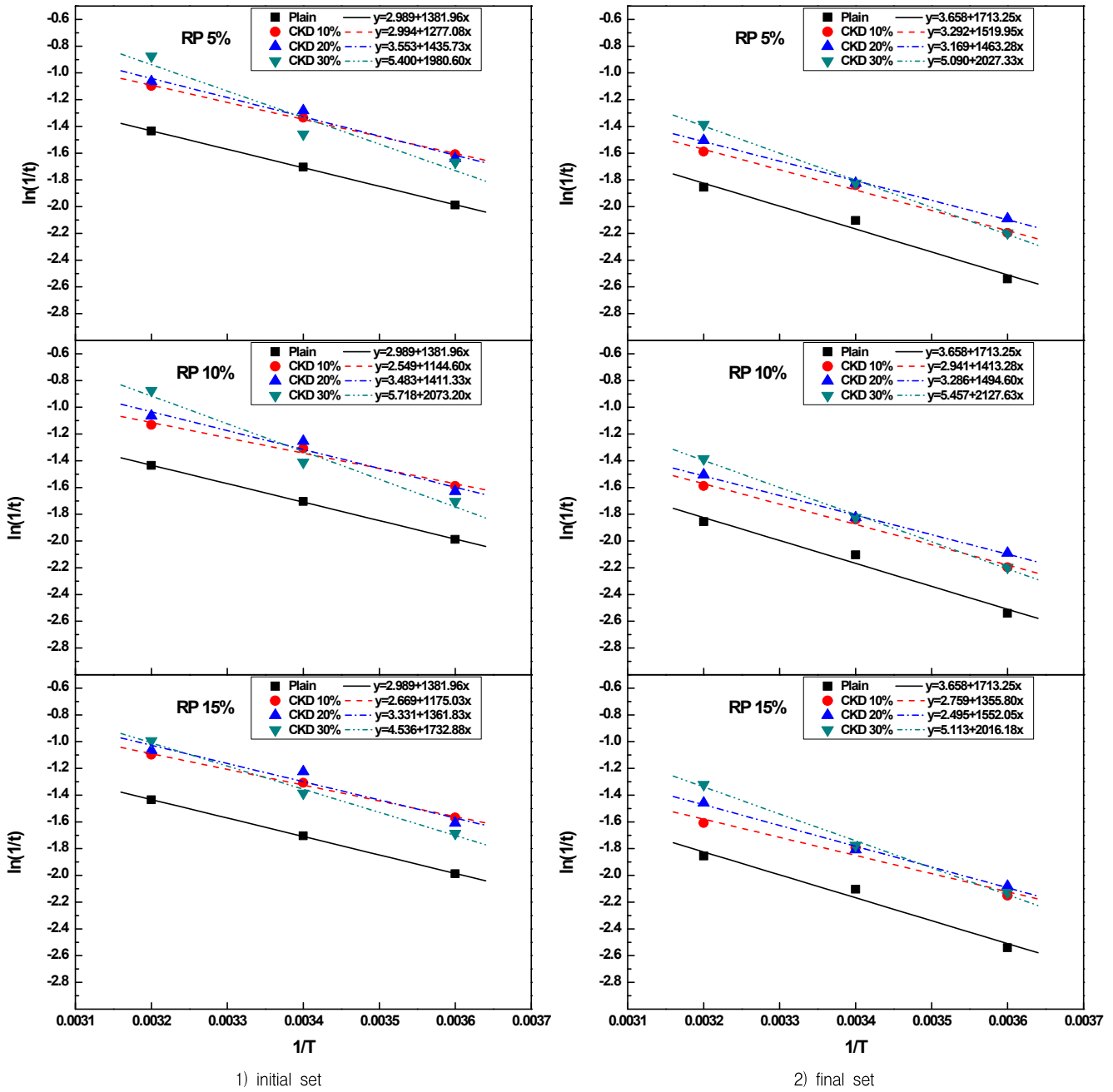


Figure 4. Arrhenius plot depending on RP and CKD contents

to the reciprocals of temperature, a finding that is similar to the results of preceding research. E_a was calculated to be 9.64 ~ 15.47 kJ/mol at initial set and 11.27 ~ 17.76 kJ/mol at final set, which is smaller than the results of around 20 kJ/mol obtained by Han et al.[11,12]. It is believed that the differences resulted from the difference in

the hydration reaction rate due to the substitution of RP and CKD.

On the other hand, Figure 5 and Table 8 show equivalent age by curing temperature calculated using E_a according to the substitution rate of PR and CKD. Overall, equivalent age by curing temperature was shown to be similar regardless of

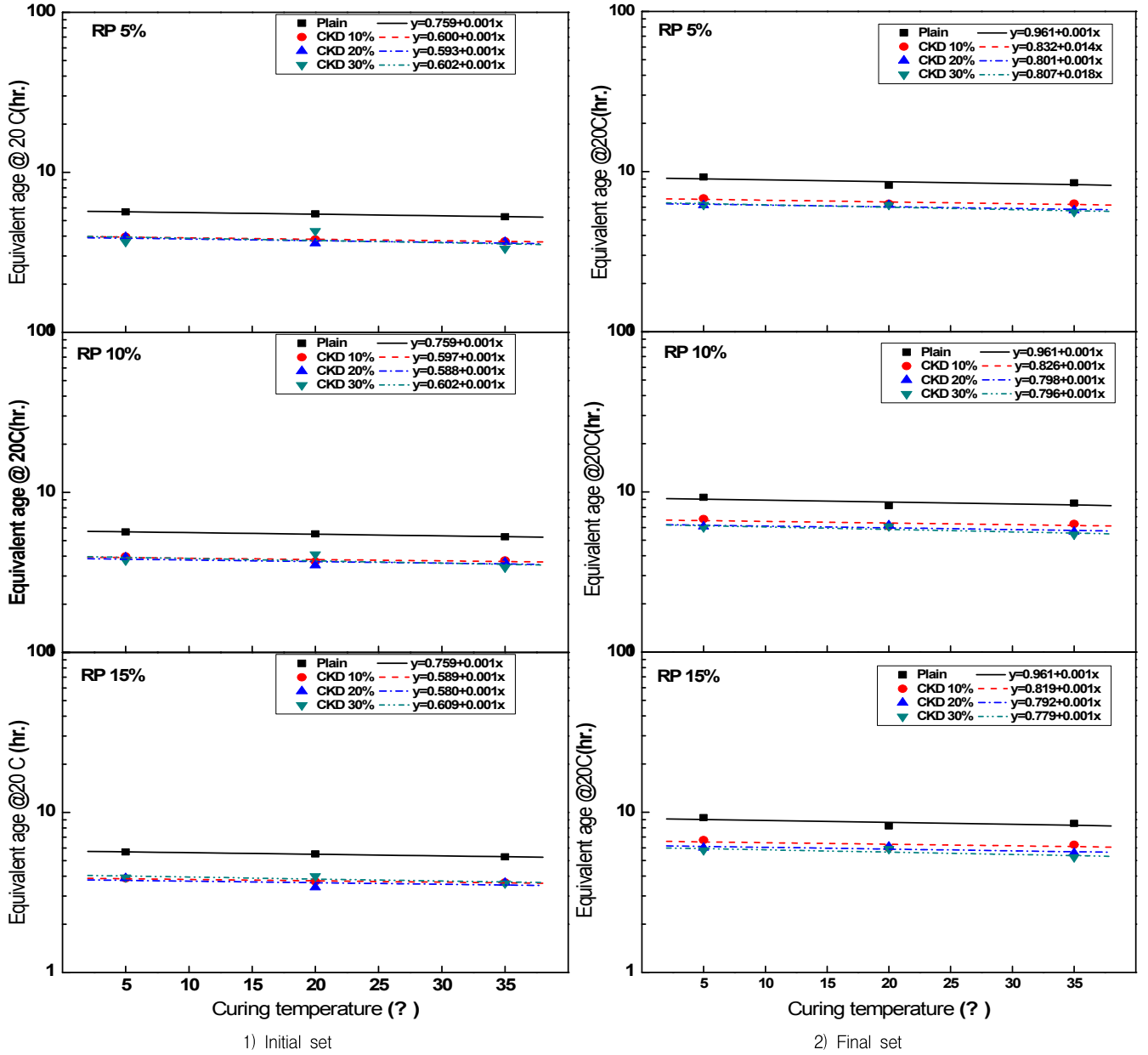


Figure 5. Relationship between equivalent age and curing temperature

the substitution rate of RP and CKD, which suggests that the setting time estimation using the equivalent age method is effective and significant based on the fact that the result satisfied the concept of equivalent age, and that the experiment result was similar to that of preceding study[11].

However, to provide a multi-regression model to

determine setting time reflecting RP and CKD based on the results from the processes above, a multiple regression/correlation analysis was conducted using the data shown in Table 9, and then Eqs. (4) and (5) were drawn, which were converted into Eqs. (6) and (7) to use equivalent day for setting time estimation.

That is, if the substitution rate of RP and CKD

and setting temperature are known, initial and final set time can be estimated using Eqs. (6) and (7).

1) Equivalent age at initial set

$$t_e = -0.049RP - 0.02CKD + 4.79 (R^2 = 0.437) \quad (4)$$

2) Equivalent age at final set

$$t_e = -0.071RP - 0.053CKD + 7.91 (R^2 = 0.734) \quad (5)$$

3) Estimating equation of initial setting time

$$t_i = \frac{-0.049RP - 0.02CKD + 4.79}{\exp\left(\frac{E_a}{R}\left(\frac{1}{T_r} - \frac{1}{T}\right)\right)} \quad \text{--- (6)}$$

4) Estimating equation of final setting time

$$t_i = \frac{-0.071RP - 0.053CKD + 7.91}{\exp\left(\frac{E_a}{R}\left(\frac{1}{T_r} - \frac{1}{T}\right)\right)} \quad \text{--- (7)}$$

Where,

t_e : equivalent age (hr.)

t_i, t_f : initial and final setting time (hr.)

RP : recycled aggregate powder (%)

CKD : cement kiln dust (%)

E_a : apparent activation energy (KJ/mol)

T : absolute temperature ($^{\circ}$ K)

Table 7. Calculation of E_a based on Arrhenius equation

RP	CKD	Setting	Regression coefficients			Ea (KJ/mol)
			$\ln(\frac{1}{t_i}) = \ln A - B \frac{1}{T}$			
			lnA	B(Ea/R)	R2	
0	0	Initial	2.989	1381.9	0.990	11.49
		Final	3.657	1713.2	0.987	14.24
5	10	Initial	2.994	1277.0	0.999	10.62
		Final	3.292	1519.9	0.995	12.64
	20	Initial	3.553	1435.7	0.989	11.94
		Final	3.169	1463.3	0.998	12.17
	30	Initial	5.400	1980.6	0.964	16.47
		Final	5.091	2027.3	0.998	16.86
10	10	Initial	2.548	1144.6	0.991	9.64
		Final	2.940	1413.2	0.985	11.75
	20	Initial	3.482	1411.3	0.994	11.73
		Final	3.286	1494.6	0.996	12.43
	30	Initial	5.718	2073.2	0.986	17.24
		Final	5.457	2127.6	0.997	17.60
15	10	Initial	2.669	1175.0	0.998	9.77
		Final	2.758	1355.8	0.982	11.27
	20	Initial	3.330	1361.8	0.972	11.32
		Final	3.494	1552.0	0.997	12.90
	30	Initial	4.536	1732.8	0.997	14.41
		Final	5.113	2016.1	0.997	16.76

Table 8. Calculation of equivalent age

RP- CKD	Curing temperatur e ($^{\circ}$ C)	Setting time(hr.)		Equivalent age @20 $^{\circ}$ C			
		Initial	Final	Initial	Average	Final	Average
Plain	5	7.30	12.70	5.66		9.26	
	20	5.50	8.20	5.50	5.48	8.20	8.66
	35	4.20	6.40	5.28		8.51	
5-10	5	5.00	9.00	3.95		6.80	
	20	3.80	6.30	3.80	3.82	6.30	6.47
	35	3.00	4.90	3.71		6.31	
5-20	5	5.15	8.08	3.95		6.17	
	20	3.60	6.20	3.60	3.75	6.20	6.04
	35	2.90	4.50	3.68		5.74	
5-30	5	5.30	9.00	3.68		6.20	
	20	4.30	6.20	4.30	3.77	6.20	6.00
	35	2.40	4.00	3.34		5.60	
10-10	5	4.90	8.80	3.97		6.78	
	20	3.70	6.10	3.70	3.81	6.10	6.40
	35	3.10	5.00	3.75		6.32	
10-20	5	5.10	8.00	3.93		6.08	
	20	3.50	6.20	3.50	3.70	6.20	5.97
	35	2.90	4.40	3.67		5.64	
10-30	5	5.50	8.90	3.75		6.01	
	20	4.10	6.10	4.10	3.75	6.10	5.84
	35	2.40	3.80	3.39		5.41	
15-10	5	4.80	8.60	3.87		6.70	
	20	3.70	6.00	3.70	3.74	6.00	6.32
	35	3.00	5.00	3.65		6.26	
15-20	5	5.00	8.00	3.89		6.01	
	20	3.40	6.10	3.40	3.64	6.10	5.89
	35	2.90	4.30	3.64		5.57	
15-30	5	5.40	8.40	3.92		5.79	
	20	4.00	5.90	4.00	3.84	5.90	5.65
	35	2.70	3.75	3.60		5.24	

Figure 6 illustrates the estimated setting time using Eqs. (6) and (7) using curing temperature according to substitution rate of RP and CKD.

Overall, the setting time became more reduced when RP and CKD were substituted. In particular, the setting time was found to be much more significantly reduced at low-temperature (5°). Based on the research findings, the initial and final set time by curing temperature according to substitution rate of RP and CKD can be estimated, enabling a more effective quality control in relation to setting reaction to be performed in the field in the future.

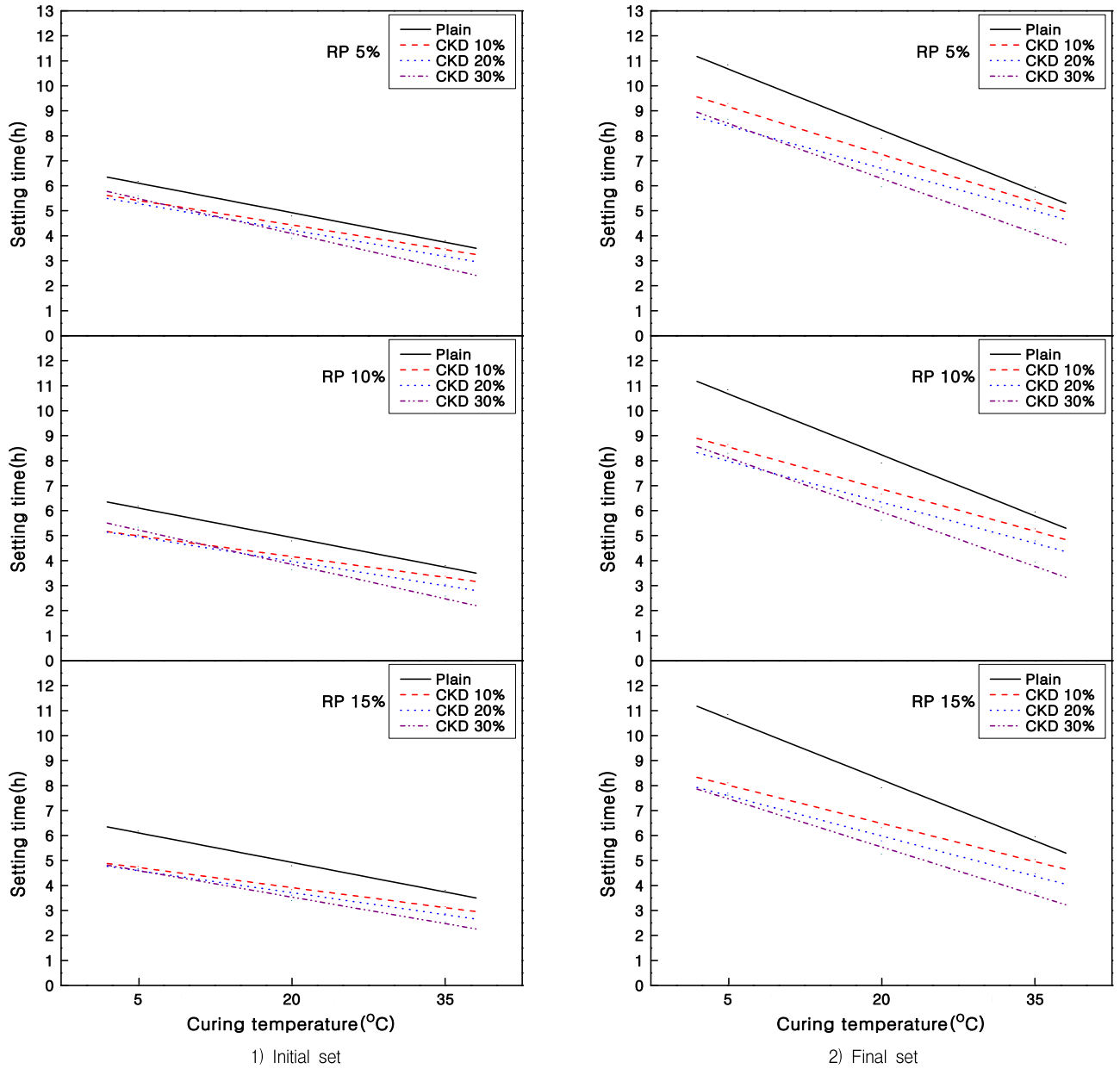


Figure 6. Relationship between curing temperature and setting time with the contents of RP and CKD

The results of a comparative analysis of the measured and estimated setting time data are shown in Figure 7. The setting time estimated in this research was shown to be similar to the measured one, on which basis the validity of the setting estimation model proposed in this research was verified. However, to perform a more accurate estimation than the model proposed in this study,

additional validation is also required by conducting a comparative analysis between the data obtained in this research and the measured setting time data using a different substitution rate of the same materials.

4. Conclusion

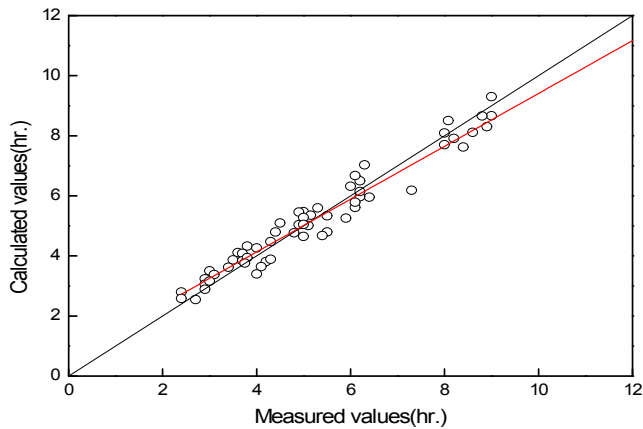


Figure 7. Comparison of measured and calculated values

This paper proposes a method of estimating the setting time of cement mortar by reflecting the substitution rate of RP and CKD under various curing conditions. The research findings are as follows.

- 1) Although the substitution rate of RP was higher, there were no significant differences in setting time, though the setting time was shown to be reduced as the substitution rate of CKD became higher. In particular, at 5°C, a low curing temperature, when the RP and CKD were mixed, the setting time was found to be much more significantly reduced than for plan mixture. When the mixture of RP and CKD is used in a low-temperature condition, the setting time is expected to be shortened.
- 2) To estimate the setting time, E_a was calculated to be 9~18 KJ/mol, E_a of cement mortar with the mixture of RP and CKD was shown to be similar to or slightly higher than that of plan cement mortar, or to be slightly lower than or similar to that of cement mortar obtained in a previous study, which found that changes in E_a are not significant according to the substitution rate of RP and CKD.
- 3) Setting time estimation model and graphs using

equivalent age of cement mortar with a mixture of RP and CKD were presented, and a high correlation was found between the actually measured data and the estimations. This is expected to serve as useful data for the effective management of the setting time in the field in the future.

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