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Article

Curing Sensitivity of Mortars Containing Cement, Calcined Clay, Fly Ash, and Limestone Powder

Cuong Manh Phung[1], **Krittiya Kaewmanee**[2,*], **and Somnuk Tangtermsirikul**[1]

1 School of Civil Engineering and Technology, Sirindhorn International Institute of Technology, Thammasat University, Pathum Thani, 12120, Thailand
2 Construction and Maintenance Technology Research Center (CONTEC), Sirindhorn International Institute of Technology, Thammasat University, Pathum Thani, 12120, Thailand
*E-mail: krittiya@siit.tu.ac.th (Corresponding author)

Abstract. The purpose of this study is to investigate the effect of multi-binder systems which consist of cement, calcined clay, fly ash, and limestone powder on the curing sensitivity of mortars. Three series of mortar with different water to binder ratios by weight (w/b) of 0.35 and 0.55 and different paste ratios of 1.2 and 1.4 were produced for testing compressive strength. The specimens of each binder system were put under two conditions of curing water curing (water-cured) and no curing (air-cured). The curing sensitivity index was calculated by considering compressive strength as an indicator. It was found that for the mixes with the same water to binder ratio and paste content, the use of fly ash increased curing sensitivity while limestone powder and calcined clay reduced curing sensitivity. Mixes with a higher water to binder ratio (w/b= 0.55) showed higher curing sensitivity compared to mixes with a lower water to binder ratio (w/b= 0.35). Reducing paste content resulted in reduced curing sensitivity of the mortars.

Keywords: Calcined clay, fly ash, limestone powder, curing sensitivity, compressive strength.

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1. Introduction

Curing of concrete is normally difficult for structures with large exposed surfaces such as slabs, pavements, walls, etc. The demands to shorten construction period is increasing. To achieve perfect curing conditions is not an easy task, especially in a hot climate country like Thailand. It is known that poor curing leads to low mechanical and durability properties.

In addition, the use of SCMs such as silica fume, rice husk ash, fly ash to partially replace cement has become a trend in the concrete manufacturing industry. Their usage is considered friendly to the environment and can enhance many performances of the concrete as well as can sometimes reduce the cost of the concrete. However, with the increasing commitments to reduce the reliance on coal-burning, most recently with the "Paris agreement on climate change", mineral admixtures from fossil fuel such as fly ash may become less abundant. Calcined clay is emerging as one of the promising materials with regards to performance and the abundant reserves [1-3]. The use of many mineral admixtures in the binder system requires different curing conditions because the curing sensitivities of concrete with different mineral admixtures are not the same. Therefore, more knowledge about the curing sensitivity of various binder systems will be of great significance. The knowledge can be applied to produce concrete with low curing sensitivity, which can be used to ease the process of curing in the actual construction, especially for concrete practice in hot climate and for structures with large exposed surface area where there are difficulties to effectively conduct the curing process.

Kinnaath et al. [4] developed a kind of concrete with a minimum curing period and introduced a concept of curing sensitivity index (CSI) for evaluation of sensitivity to curing of concrete. They found that in contrast to fly ash, limestone powder reduces the curing sensitivity of the concrete. Concrete containing both fly ash and limestone powder expressed lower curing sensitivity compared to cement-fly ash concrete. However, the behaviors and key factors related to curing sensitivity remain undetermined. Moreover, research works on curing sensitivity of systems with other mineral admixtures are still very limited. This paper focuses primarily on investigating the behavior of different binder systems with cement, calcined clay, fly ash and limestone powder on curing sensitivity. Two vital parameters, which are paste content and water to binder ratio, were studied. Normal consistency, setting time, flow, and compressive strength were tested and curing sensitivity of mortars by considering compressive strength was determined. Finally, the relationship between the compressive strength ratio and curing sensitivity index was presented.

2. Materials and Methods

2.1. Materials

An ordinary Portland cement (OPC) produced by Tung Song cement plant in Southern Thailand was used. This cement is an ordinary Portland cement Type I according to ASTM-C150 and TIS- 15 [5, 6]. Calcined clay (CC), fly ash (FA), and limestone powder (LP) were used to partially replace cement. Physical properties and chemical compositions of the binders are shown in Table 1. The calcined clay and limestone powder were produced by Siam Cement Group Co.Ltd. (SCG), Thailand. The mean particle size of calcined clay and limestone powder used in the test were 17 and 10 microns, respectively. The fly ash, from the Mae Moh power plant in the north of Thailand, is classified as class C according to ASTM C618 [7] and class 2b according to TIS 2135 [8]. The particle shapes of binders, analyzed by Scanning Electron Microscope (SEM), are shown in Fig. 1. Natural river sand was used as fine aggregate. The natural river sand had a specific gravity of 2.6 at the saturated surface dry condition and an absorption of 1.1%.

2.2. Experimental Design and Sample Preparations

2.2.1. Mix parameters and curing conditions

Eleven binder systems of calcined clay, fly ash, and limestone powder with different percentage of replacement by weight of total binder are designed to compare the effects of the binder systems on properties of pastes and mortars (see Table 2).

Mix proportions were designed by varying the ratio of paste volume to void volume of compacted aggregate phase (γ) and water to binder ratio (w/b). The ratio of paste volume to void volume of compacted aggregate phase (γ) is defined as

$$\gamma = \frac{V_p}{V_{void}} \tag{1}$$

where V_p is the volume of paste in a unite volume of mortar (m^3) and V_{void} is the volume of the void in the densely compacted fine aggregate in a unit bulk volume (m^3). The volume of paste can be derived as,

$$V_p = V_c + V_{cc} + V_{fa} + V_{lp} + V_w + V_{air} \tag{2}$$

where V_c, V_{cc}, V_{fa}, V_{lp}, V_w and V_{air} are the volume of cement, calcined clay, fly ash, limestone powder, water and air, respectively, in a unit volume of mortar mixture (m^3).

Table 1. Chemical compositions and physical properties of ordinary Portland cement type I, limestone powder, calcined clay, and Mae Moh fly ash.

Properties	**Ordinary Portland cement type I**	**Limestone powder**	**Calcined clay**	**Mae Moh Fly ash**
Chemical Composition (%):				
SiO_2	19.31	0.79	46.75	36.18
Al_2O_3	5.30	0.42	24.97	20.21
Fe_2O_3	3.24	0.23	11.58	13.89
CaO	63.19	54.66	5.69	18.74
MgO	2.38	0.59	3.20	2.69
SO_3	3.03	-	0.04	3.74
Na_2O	0.02	0.02	1.25	1.14
K_2O	0.92	0.01	0.53	2.29
TiO_2	0.24	0.01	2.07	0.40
P_2O_5	0.03	0.03	0.30	0.21
MnO	0.03	0.01	0.10	0.12
LOI	2.23	43.21	3.33	0.25
Specific gravity	3.05	2.57	2.67	2.57
Blaine fineness (cm^2/g)	3141	3955	3196	2254

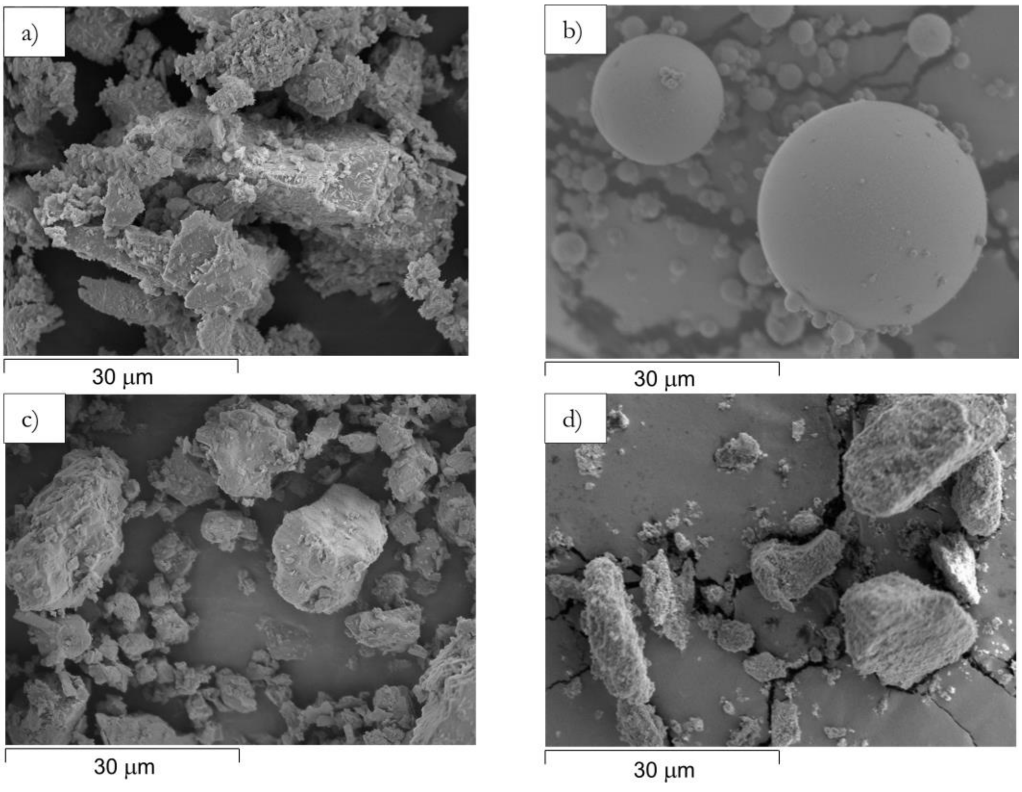

Fig. 1. Particle shape of materials by SEM: a) OPC type I; b) Mae Moh fly ash; c) Limestone powder; d) Calcined clay.

In this research, there were three series of mixtures i.e. Series A (w/b= 0.35 and γ=1.4), Series B (w/b=0.55 and γ=1.4), and Series C (w/b=0.55 and γ=1.2). The binder systems were varied in the same pattern for all series. This experimental design enables the authors to compare the effects of water to binder ratios (low w/b of 0.35 and high w/b of 0.55), paste contents (low paste content, γ=1.2, and high paste content, γ=1.4) and behavior of binder systems on compressive strength and curing sensitivity index of the mortars. Details of all mix proportions are shown in Table 3. All mixes were exposed to two different curing conditions, which were water curing (WC) and no curing or air curing (NC) in a control room with temperature and RH of 28 ± 2°C and 80 ± 5%, respectively. Compressive strength was tested at 7, 28 and 91 days of age. The compressive strength of each mix with the same curing condition is computed from the average of the three samples.

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Table 2. The tested binder systems and normal consistency.

No.	Mix designation	Replacement percentage (%)				Normal Consistency (%)
		OPC	LP	CC	FA	
1	OPC	100	-	-	-	24.04
2	10LP	90	10	-	-	23.67
3	20LP	80	20	-	-	23.48
4	10CC	90	-	10	-	25.07
5	20CC	80	-	20	-	28.00
6	30CC	70	-	30	-	29.50
7	30FA	70	-	-	30	20.45
8	20FA10LP	70	10	-	20	21.08
9	20CC10LP	70	10	20	-	27.57
10	20CC10FA	70	-	20	10	26.50
11	13CC7LP10FA	70	7	13	10	25.11

Note: The weight of the binder for each batch of mixing is 650 grams. (%) is the replacement percentage by weight.

Table 3. Mix proportions for compressive strength test (Series A, B, and C).

No.	Mix designation	w/b	γ	OPC (kg/m^3)	LP (kg/m^3)	CC (kg/m^3)	FA (kg/m^3)	Water (kg/m^3)	Sand (kg/m^3)
Series A									
1	OPC	0.35	1.4	750	0	0	0	262	1274
2	10LP	0.35	1.4	669	74	0	0	260	1274
3	20LP	0.35	1.4	589	147	0	0	258	1274
4	10CC	0.35	1.4	670	0	75	0	261	1274
5	20CC	0.35	1.4	592	0	148	0	259	1274
6	30CC	0.35	1.4	514	0	220	0	257	1274
7	30FA	0.35	1.4	511	0	0	219	256	1274
8	20FA10LP	0.35	1.4	511	73	0	146	256	1274
9	20CC10LP	0.35	1.4	513	73	147	0	257	1274
10	20CC10FA	0.35	1.4	513	0	147	73	257	1274
11	13CC7LP10FA	0.35	1.4	512	49	98	73	256	1274
Series B									
12	OPC	0.55	1.4	579	0	0	0	318	1274
13	10LP	0.55	1.4	517	58	0	0	316	1274
14	20LP	0.55	1.4	457	114	0	0	314	1274
15	10CC	0.55	1.4	518	0	58	0	317	1274
16	20CC	0.55	1.4	458	0	115	0	315	1274
17	30CC	0.55	1.4	399	0	171	0	313	1274
18	30FA	0.55	1.4	397	0	0	170	312	1274
19	20FA10LP	0.55	1.4	397	57	0	113	312	1274
20	20CC10LP	0.55	1.4	398	57	114	0	313	1274
21	20CC10FA	0.55	1.4	398	0	114	57	313	1274
22	13CC7LP10FA	0.55	1.4	398	38	76	57	313	1274
Series C									
23	OPC	0.55	1.2	496	0	0	0	273	1462
24	10LP	0.55	1.2	444	49	0	0	271	1462
25	20LP	0.55	1.2	392	98	0	0	269	1462
26	10CC	0.55	1.2	444	0	49	0	272	1462
27	20CC	0.55	1.2	393	0	98	0	270	1462
28	30CC	0.55	1.2	341	0	147	0	269	1462
29	30FA	0.55	1.2	340	0	0	146	267	1462
30	20FA10LP	0.55	1.2	340	49	0	97	267	1462
31	20CC10LP	0.55	1.2	341	49	98	0	268	1462
32	20CC10FA	0.55	1.2	341	0	98	49	268	1462
33	13CC7LP10FA	0.55	1.2	341	33	65	49	268	1462

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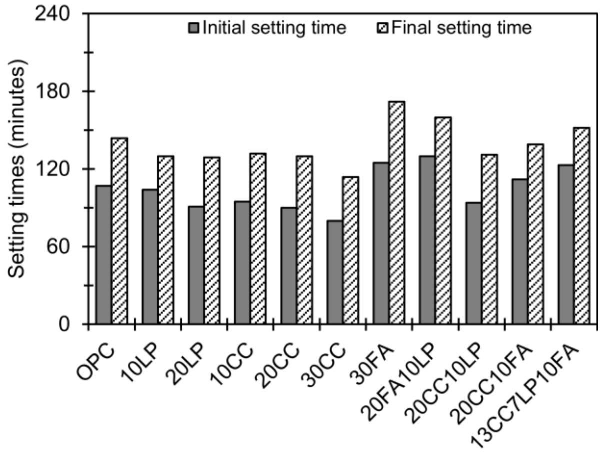

Fig. 2. Setting times of all tested binder systems.

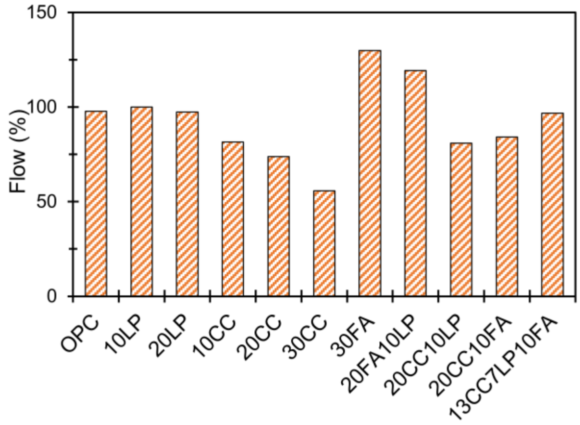

Fig. 3. Flow of all tested mortars with w/b=0.55, γ=1.2.

2.2.2. Experimental methods

The normal consistency and setting times of pastes with different binder systems were determined using the Vicat apparatus according to ASTM-C187 [9], and ASTM-C191 [10], respectively.

Flow of the mortars, following ASTM-C1437 [11], was measured immediately after mixing. Mortar cubes with dimensions of 50mm x 50 mm x 50 mm were cast to determine the compressive strength, according to ASTM-C109 [12].

Strength activity index ($SAI_{fc'}$) of mortars at the same curing conditions was calculated using Eq. (3).

$$SAI_{fc'} = \frac{A}{B} \times 100\% \quad (3)$$

where A is the compressive strength of the considered mortar. B is the compressive strength of the reference cement-only mortar.

The curing sensitivity index of mortars by considering compressive strength ($CSI_{fc'}$) was calculated using Eq. (4). The index is the different percentage between the compressive strength of a water-cured mortar and that of the no-cured (or air-cured) mortar with the same mixture.

$$CSI_{fc',t} = \frac{(fc'(t)_{WC} - fc'(t)_{NC})}{fc'(t)_{WC}} \times 100\% \quad (4)$$

where $fc'(t)_{WC}$ and $fc'(t)_{NC}$ are the compressive strengths of water-cured and no-cured (or air-cured) specimens at t days of ages, respectively. The $CSI_{fc',t}$ value is obtained from the average of three specimens with the standard deviation of less than 5%.

3. Results and Discussions

3.1. Normal Consistency and Setting Times

The normal consistency and setting times of the tested pastes are shown in Table 2 and Fig. 2, respectively. The normal consistency of the limestone powder pastes was slightly smaller than the normal consistency of the cement-only paste meaning that the limestone powder pastes required less water for a specified consistency. The calcined clay pastes required more water than the cement-only paste. Normal consistency also increased when increasing the amount of calcined clay as the water requirement of calcined clay is high due partly to the rough surface of calcined clay particles [13]. The fly ash pastes had the lowest normal consistency. The results of ternary and quaternary binder systems showed that their normal consistencies depend on the water requirement and content of the replacing materials.

As shown in Fig. 2, the setting times of all binder systems satisfy the requirements of ASTM C150 [5] (initial setting time not earlier than 45 min but not longer than 375 min for final setting time). The limestone powder pastes and calcined clay pastes show shorter setting times than the cement-only paste. The higher the limestone powder and calcined clay contents, the shorter the setting times. The results can be explained by that limestone powder accelerates the hydration of cement [14]. Calcined clay is a supplementary cementitious material (SCM) with high content of alumino-silicate phases, so it hardens rapidly in the presence of water [15-17]. The 30FA paste demonstrates the longest setting times. It is well known that the use of fly ash can delay the setting times due to the cement dilution and slow pozzolanic reaction [18].

3.2. Flow

The flow results of all tested mortar mixtures are shown in Fig. 3. According to ASTM-C1437 [11], the flow is the resulting increase in average base diameter, expressed as a percentage of the original base diameter (100 mm).

The flow of mortar with 30% fly ash is the largest, due to the spherical shape of fly ash particles (Fig. 1(b)). The effects of limestone powder on flow of mortars are insignificant. The mixtures with calcined clay show lower flow compared to all other binder systems. The flow decreases when increasing replacement percentage of

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calcined clay from 10% to 30%. It is known that water requirement of calcined clay is high, resulting in lower workability of the mixtures when calcined clay is incorporated into the mixtures [19].

Ternary binder systems exhibit better flow for the mixes containing fly ash (20FA10LP and 20CC10FA) than their respective binary mixtures without fly ash. The quaternary binder system (13CC7LP10FA) shows similar flow to the control mix. Combining calcined clay with fly ash or limestone powder or both fly ash and limestone powder helps to balance out the negative impact of calcined clay, thus decreases the water requirement when compared to the binary mixtures with only calcined clay.

3.3. Effect of Binder Systems on Strength Activity Index

The effect of binder systems on strength activity index for different water to binder ratios and paste contents are shown in Fig. 4, Fig. 5, and Fig. 6 for tested mixtures in series A, B, and C, respectively. In the case of binary binder systems, mortars with 10% and 20% limestone powder show lower strength activity index than the control mix at all ages for all tested w/b, paste contents, and curing conditions. The strength activity index of the limestone powder mortars is lower when increasing limestone powder replacement in the binder system, mainly due to lower cement content.

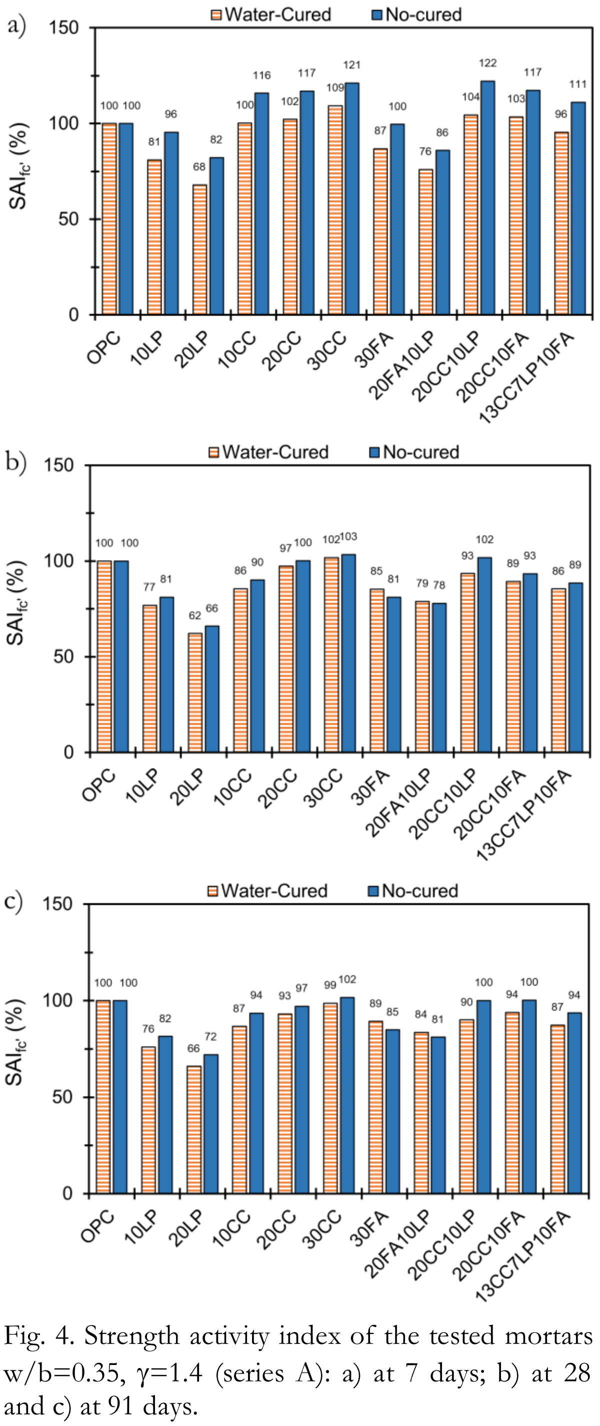

Fig. 4. Strength activity index of the tested mortars with w/b=0.35, γ=1.4 (series A): a) at 7 days; b) at 28 days and c) at 91 days.

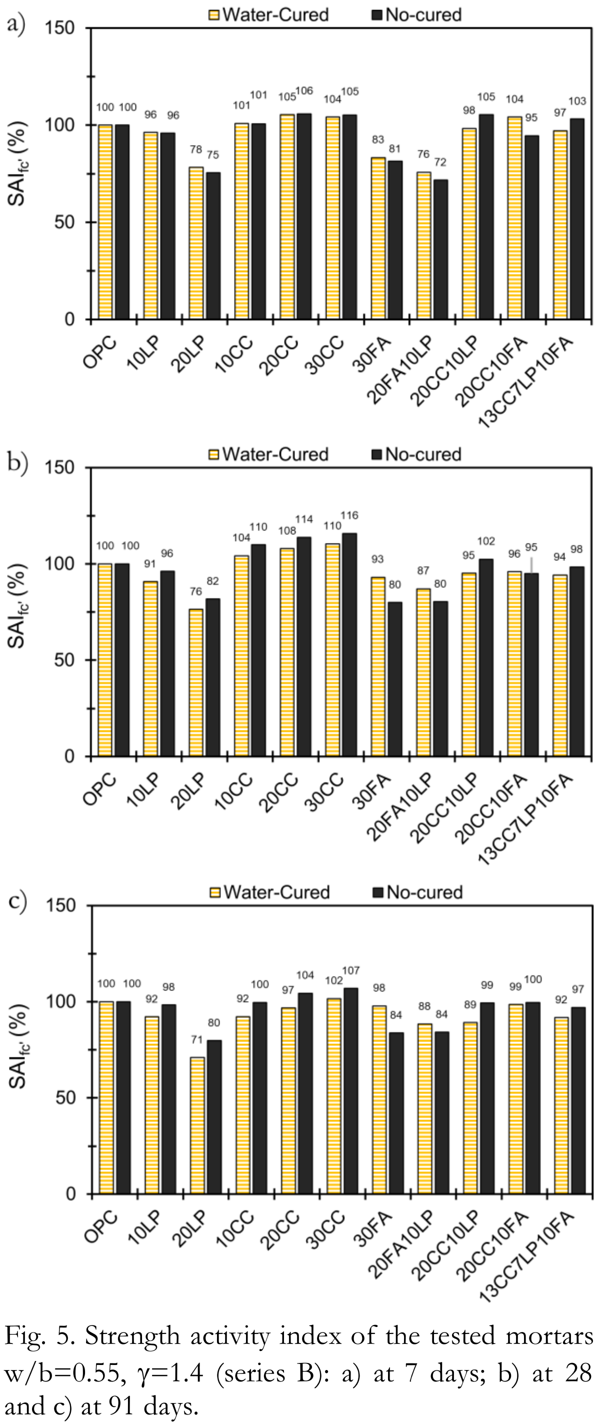

Fig. 5. Strength activity index of the tested mortars with w/b=0.55, γ=1.4 (series B): a) at 7 days; b) at 28 days and c) at 91 days.

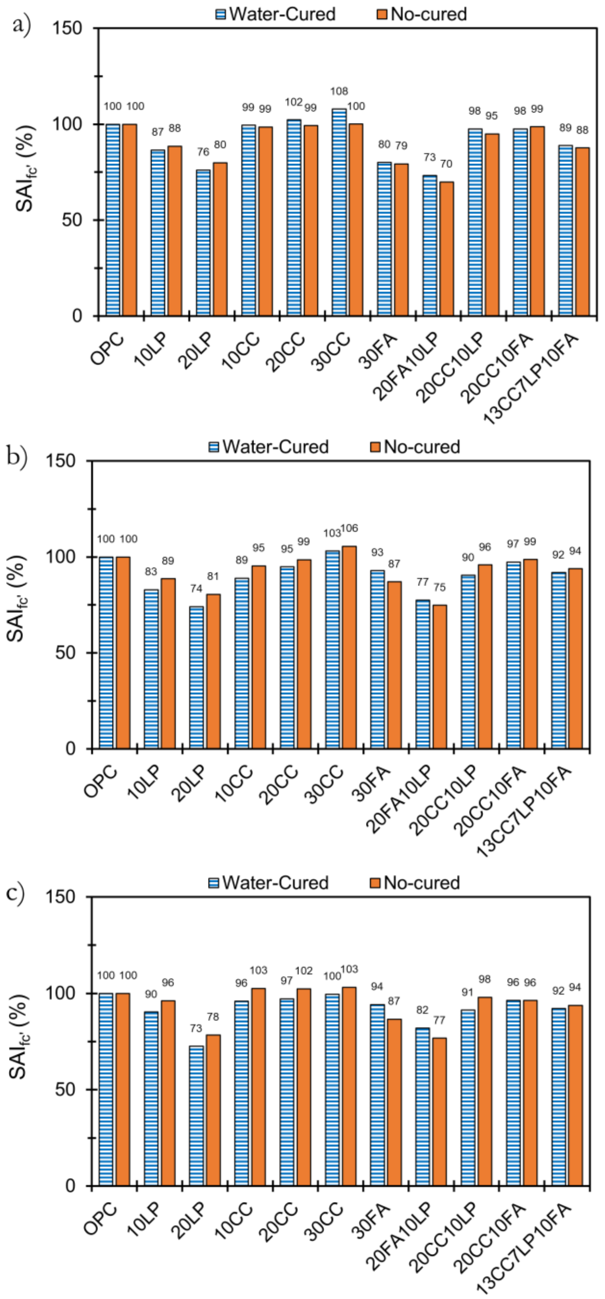

Fig. 6. Strength activity index of the tested mortars with w/b=0.55, γ=1.2 (series C): a) at 7 days; b) at 28 days and c) at 91 days.

For mortar with 10%, 20%, and 30% calcined clay, higher strength activity index at 7days than the control mix in all series and curing conditions are observed. At 28 days, the strength activity indices of all calcined clay mixtures in series B are higher than that of the control mix. In other series, only the 30CC mix exhibits higher strength activity index than the control mix (OPC). At 91 days, the strength activity indices of all mixes are lower than control mix, except 30CC mix in series B. For the mix with 30% fly ash, the strength activity index is lower than control mix in all series and curing conditions.

In the case of ternary binder systems, mortars with 20CC10LP and 20CC10FA show high strength activity index, whereas mortar with 20FA10LP demonstrates the lowest strength activity index. For quaternary mix, 13CC7LP10FA exhibits lower strength activity index than the control mix.

3.4. Curing Sensitivity

3.4.1. Effect of paste contents on curing sensitivity index

Figure 7 presents the effects of paste content (γ=1.2 and γ=1.4) on $CSI_{fc'}$ of mortars with w/b of 0.55 at 28 and 91 days. It was found that lower paste content helps to reduce curing sensitivity index for all binder systems. This is because curing affects only paste but not aggregates.

3.4.2. Effect of water to binder ratio on curing sensitivity index

Effects of water to binder ratio (w/b=0.35 and 0.55) on $CSI_{fc'}$ of mortars with γ of 1.4 at 28 and 91 days are shown in Fig. 8. Higher water to binder ratio leads to higher $CSI_{fc'}$ in all cases. This is because a higher w/b mortar is more porous and so more subjective to moisture loss (drying) when curing is not sufficiently provided (poor curing).

3.4.3. Effect of binder systems on curing sensitivity index

Figure 7 and 8 show the effects of binder system on $CSI_{fc'}$ at 28 and 91 days of all mortars series. For the binary binder systems, mortars with limestone powder have lower curing sensitivity than the control mix (OPC), and $CSI_{fc'}$ reduces at higher limestone powder replacement. Calcined clay can help to reduce curing sensitivity at all tested replacements (10%, 20%, and 30%). However, $CSI_{fc'}$ of a mortar increases with increasing calcined clay content, which is opposite to the limestone powder case. The mix with 30% fly ash has the highest $CSI_{fc'}$. The tendency is similar for all series. It can be explained by the fact that limestone powder is a filler material, so there is no reaction with water, however, it can accelerate the hydration of cement especially at early ages [14, 20, 21]. Many studies demonstrate that calcined clay behaves similarly to many supplementary cementitious materials (SCMs) and a large portion of calcined clay reacts at early ages [15-17]. The fast reaction of the calcined clay can be seen from the results of heat evolution tested in a calorimeter of pastes with 30% calcined clay compared to cement-only paste as shown in Fig. 9. The above explain the mechanism that helps to reduce $CSI_{fc'}$ of the calcined clay mixes. In contrast, fly ash starts to have pozzolanic reaction at later ages. It requires longer curing period, thus leading to a higher $CSI_{fc'}$ [4, 18].

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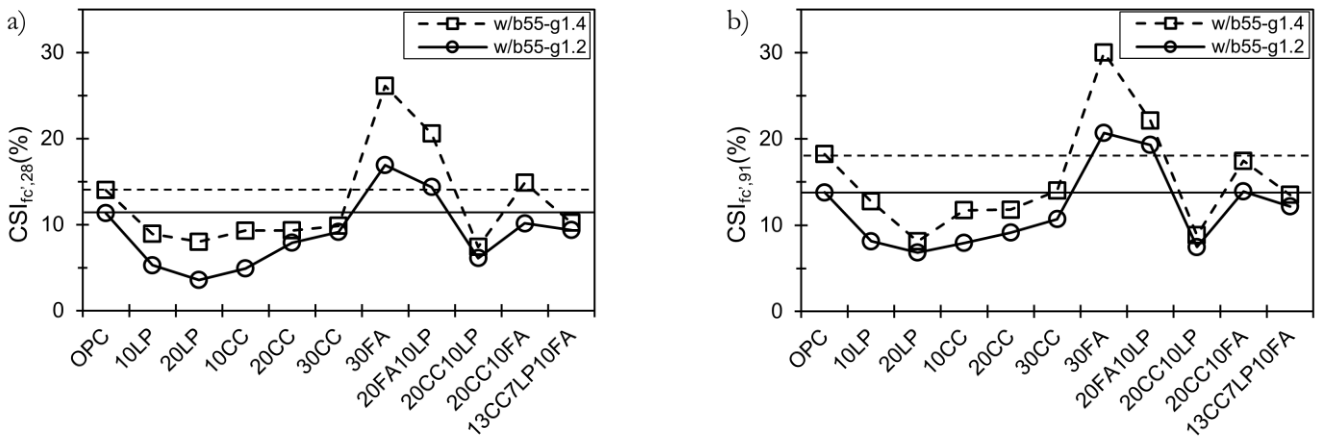

Fig. 7. Curing sensitivity index of tested mortars by compressive strength with different paste content: a) $CSI_{fc'}$ at 28 days; b) $CSI_{fc'}$ at 91 days.

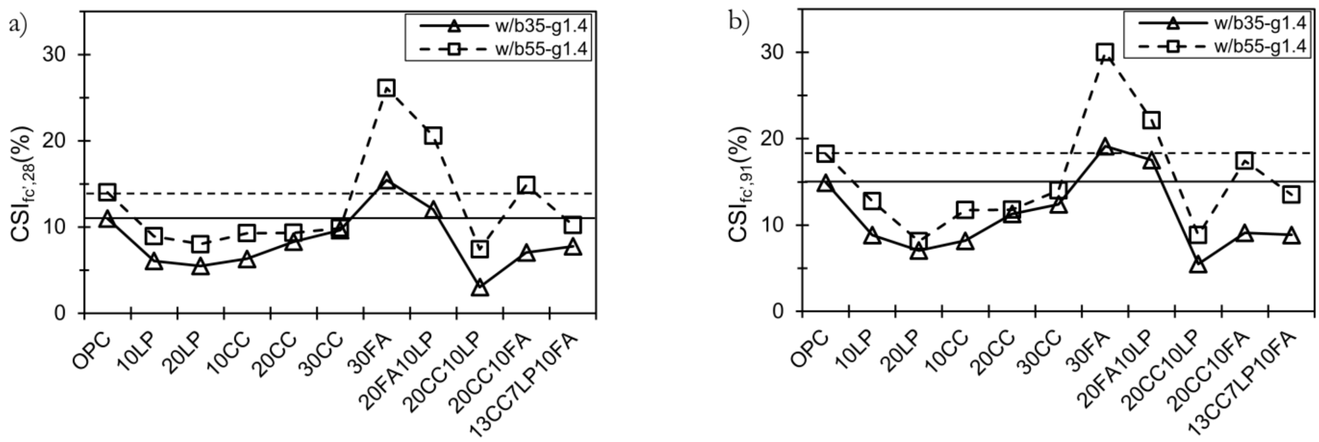

Fig. 8. Curing sensitivity index of tested mortars by compressive strength with different water to binder ratio: a) $CSI_{fc'}$ at 28 days; b) $CSI_{fc'}$ at 91 days.

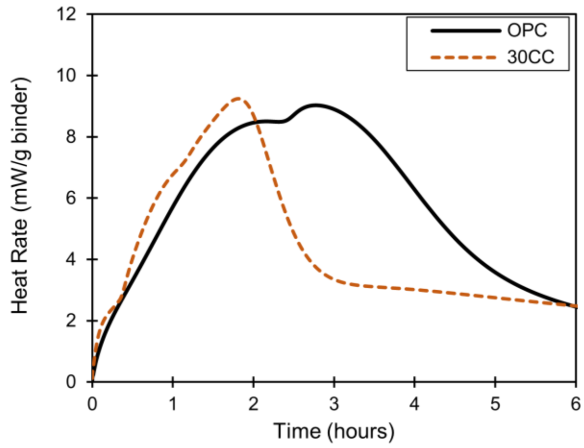

Fig. 9. Heat rate of cement-only paste and paste with 30 % of calcined clay replacement.

For ternary and quaternary binder systems with the same replacement percentage of SCM (30%), 20FA10LP has the highest $CSI_{fc'}$ while the 20CC10LP has the lowest curing sensitivity index. Mixes containing fly ash have higher $CSI_{fc'}$ than mixes without fly ash.

3.4.4. Relationships between setting times and curing sensitivity index

Figure 10 shows the relationships between the $CSI_{fc'}$ of mortars (series B: w/b= 0.55, γ= 1.4) at 91 days and setting times of their corresponding pastes. The mortars of series B with the highest paste content and water to binder ratio were considered. Although the regressive relationship between $CSI_{fc'}$ and setting times have low coefficient of determination (R^2) and high mean absolute errors (MAE), there is a tendency that the shorter setting times leads to the lower $CSI_{fc'}$ of the mortars.The main reason is that setting times have no direct relationship with strength. For initial setting time, ettringite formation but not the development of hydration or pozzolanic reaction products such as C-S-H, control the initial setting of the mixtures, and so the mixtures are not in the hardened state yet. For final setting time, the cementitious paste just begins to harden, reaction to produce products which generate strength just started. These cause the low accuracies of the correlations between setting times and curing sensitivity index which is computed based on strength.

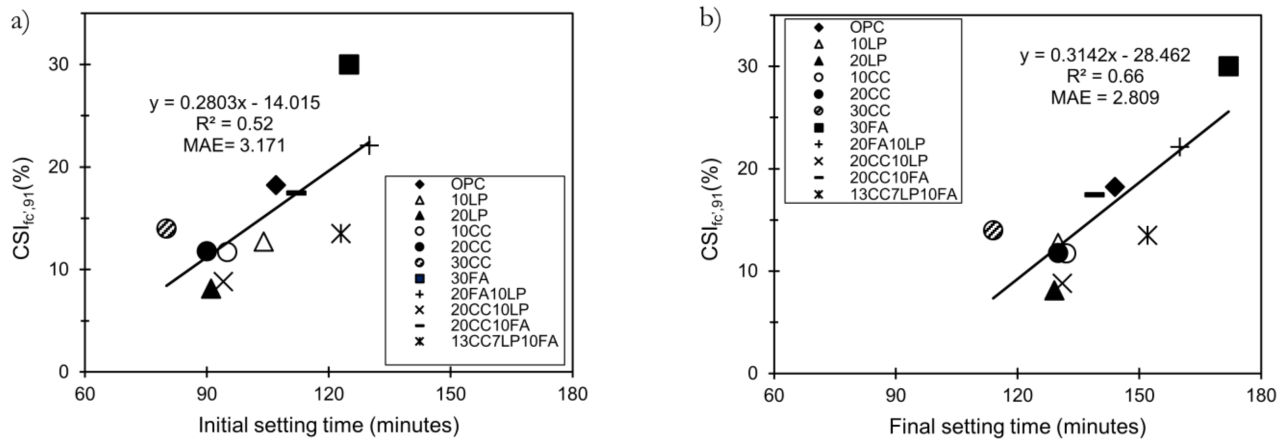

Fig. 10. Relationships between setting times and $CSI_{fc'}$ at 91 days of tested mortars with w/b= 0.55, γ= 1.4: a) $CSI_{fc'}$ at 91 days versus initial setting time; b) $CSI_{fc'}$ at 91 days versus final setting time.

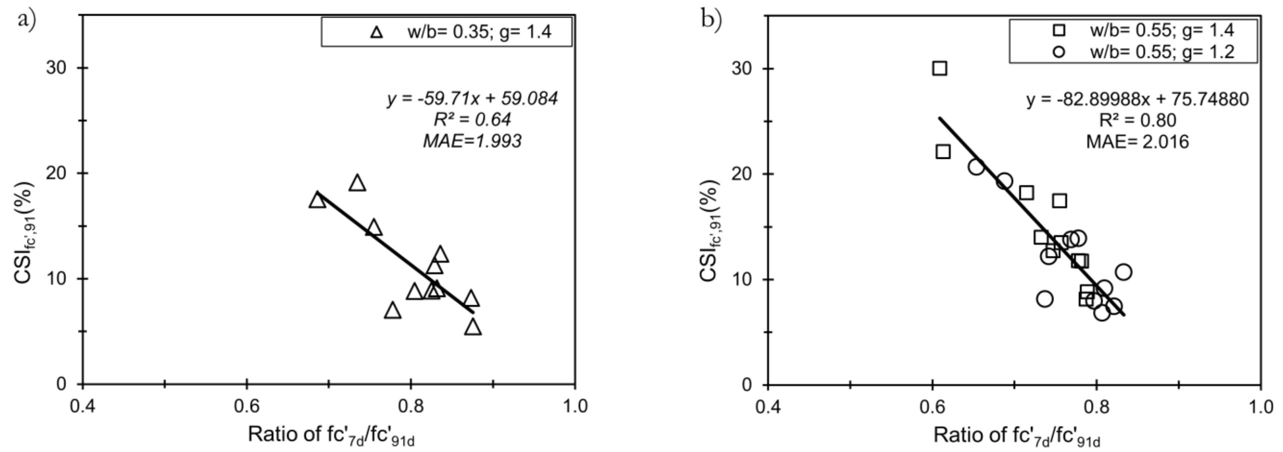

Fig. 11. Relationships between compressive strength ratios (fc' at 7d/fc' at 91d) and $CSI_{fc'}$ at 91 days of the tested mortars: a) at water to binder ratio of 0.35; b) at water to binder ratio of 0.55.

3.4.5. Relationships between ratio of early age strength to ultimate strength and curing sensitivity index

Figure 11 presents the relationship between ratio of early age strength (at 7 days of age) to ultimate strength (defined here at 91 days of age) and $CSI_{fc'}$ at 91 days of the tested low water to binder (w/b= 0.35) and high water to binder (w/b= 0.55) mortars. It was found that $CSI_{fc'}$ decreases with an increase in ratio of early age strength to ultimate strength. The correlation between ratio of early age strength to ultimate strength and $CSI_{fc'}$ generally exhibits a linear trend.

The relationship of the high water to binder (w/b= 0.55) mixes shows a coefficient of determination (R^2) and mean absolute errors (MAE) of 0.80 and 2.016%, respectively. While low water to binder (w/b = 0.35) mixes have R^2 and MAE of 0.64 and 1.993%, respectively. By comparing Fig. 10 with Fig. 11, it can be said that curing sensitivity index has higher correlation with the compressive strength ratio, indicated by the ratio of 7-day compressive strength to 91-day compressive strength, than the setting times. From these results, the compressive strength ratio may be useful as an indicator for evaluation of curing sensitivity of concrete with various binder systems.

4. Conclusions

Curing sensitivity is greatly affected by paste content, water to binder ratio, binder types and binder systems. Mixtures with lower water to binder ratios and lower paste contents resulted in lower curing sensitivity. In addition, binder systems with calcined clay and limestone powder, which shorten setting times, reduce the curing sensitivity index of mortars. Although there is a relationship between setting times and curing sensitivity index, the accuracy is unsatisfied. On the other hand, ratio of early age strength to ultimate strength showed better correlation with curing sensitivity index such that the higher the ratio of early age strength to ultimate strength, the lower the curing sensitivity index. This ratio can be an indication factor to determine the curing sensitivity of concrete.

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