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Effect of Clinker Phase Changes on Cement Performance and its Reactivity with Super Plasticizer Additive

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ABSTRACT

Based on various sources and experiences, the main phases of clinker significantly impact the technical characteristics of cement and its performance in the resulting concrete. The interaction of clinker phases with concrete admixtures is also obvious, but the effect of each phase and their optimal values for optimal concrete performance is complicated. Therefore, in this study, this issue was an extensive laboratory operation was designed. So 11 samples were selected whose phase values were varied. Then laboratory cement was performed under the same conditions using 11 clinker samples, and physical and chemical analysis of cement samples was performed. So 22mixtures were prepared so that 11mixtures with the title of control and 11mixtures with the title of additives. Based on the results, comparing the effect of C3S clinker on the compressive strength of standard mortar and concrete showed that there is no direct correlation between C3S and compressive strength, but it can be said that with each percentage increase of C3S clinker, about 1.5 unit (kg/cm2). However, with the increase of C3S from about 57% onwards, the decrease in concrete strength is clearly seen, which can be referred to as the negative effect of excessive C3S phase (due to the solubility of concrete and increasing the w/c ratio). Based on the results of air determination experiments of fresh concrete mixtures containing additives and its declining trend in this study, it was found that the optimal value of C3A is about 5-6%.

Keywords: Alite, Aluminate, Additive.

1. INTRODUCTION

Concrete industry experts often prefer that the cement used contains a large amount of Alite phase (C3S) to ensure that the strength and durability of the concrete are easily achieved. On the other hand, increasing the Alite phase can have adverse effects on cement granulation, fresh concrete performance, the durability of hardened concrete, and other parameters [1]. According to Hills studies (2007), aluminate contributes to the strength of cement paste at 1 to 3 days of age (and possibly longer). The role of alumina in the strength of standard mortar and concrete is controversial, as some sources have explicitly stated that increasing the short-term compressive strength of alumina increases the short-term strength of standard mortar and concrete, but others have stated that alumina is practically insignificant in terms of compressive strength. However, according to the diagram in Figure 1, the effect of the Alite phase on the acquisition of short and medium-term strength is quite clear [2-7]. However, it seems that increasing the aluminate and Alite phases increases the water demand of concrete. Probably due to this increase in water demand in the production of traditional concrete, which increases the concrete water to achieve the desired performance, a decrease in the compressive strength of concrete occurs.

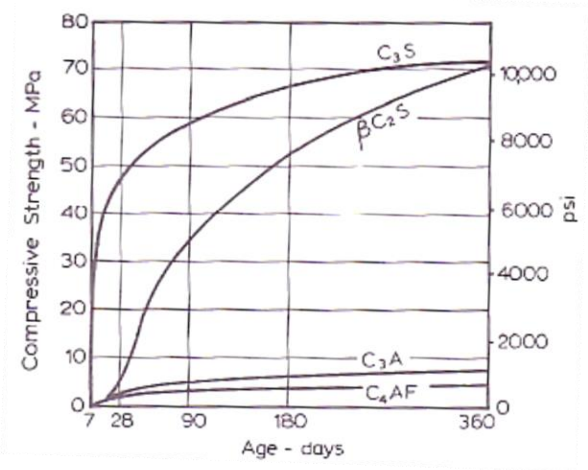


Figure 1. Process of achieve strength of clinker phases during time [8-9].

According to various sources, the hydration of Belite is slower than the Alite [10-12]; therefore, the process of achieving the strength of concrete obtained from cement with high Belite is relatively slow. Table 1 shows the hydration Alite and Belite during the time [13-15].

Table 1. Degree of hydration of Alite and Belite [13-15]

Heat hydration		Depth of hydration (microns) after			Degree of hydration (%) after		phases
(Calories per gram)	(Joules per gram)	180days	28days	3days	28days	1day	
120	500	15.0	7.9	3.5	78-80	25-35	**Alite (C3S)**
60	250	2.7	1.0	0.6	20-50	5-10	**Belite (C2S)**

Lin et al. (2018) studied the relationship between concrete strength at different ages and cement. By selecting type 1-425 and 2 as the most widely used cement in Iran and making concrete with a grade of 350 kg/m3 as the most widely used grade of concrete with equal and psychological materials and processing under the same conditions, they try to evaluate, analyze and compare simultaneously. The results had compressive strength [16]. Specialists conducted similar studies to evaluate the effect of the main phases of cement on initial and long-term strength [17-19]. Based on the results of these studies, the amount of C3A phase in the early strength, the amount of C3S phase in the compressive strength of tests of all ages have favorable effects. Also, the number of alkalis has a clear effect on the growth of resistance. Rabiee et al. (2007) in a study investigated the effect of liquid to solid phase ratio on the compressive strength of standard cement mortar. During four series of laboratory work, they adjusted the values of this ratio (liquid phase to solid phase or L/P) 0.30, 0.35, 0.40, and 0.45 and measured the compressive strength of the 28-day standard mortar. According to the results of these researchers, with increasing the liquid phase, the 28-day compressive strength decreases, and with the addition of aqueous monosodium phosphate, this compressive strength decreases [20]. Aghabgloo et al. (2017) investigated the effect of aluminate content (C3A) on the properties of cement paste, mortar, and concrete mixtures containing additives. They made three concrete mixtures using three Portland commercial cement of type 1-425 with different C3A content (1.90%, 2.77%, and 5.85%) and studied the rheological properties of fresh concrete and its compressive strength. According to the results of these researchers, by reducing the C3A of cement, the rheological properties of fresh concrete and the durability of the consistency of the mixtures are improved, but the compressive strength of concrete mixtures decreases at an early age. They proved that with the increase of cement aluminate, the need for water-reducing additive increases in the same flow. Based on these results, in relatively low aluminate cement compared to relatively higher aluminate cement: 1-day strength loss of about 40%; 3 days is about 10% and at 7 and 28 days is less than 5%. According to this study, the psychological decline of mortar and concrete is improved by reducing aluminate [21]. Erdogan (2013) investigated the effect of clinker phase distribution in cement particles on the properties of hydrated cement paste. He analyzed SEM/X-ray images of laboratory cement and cement and concrete experiments and used them in the simulation to investigate the effect of the spatial distribution of clinker phases on particles on hydration and the resulting properties. According to the results of this researcher, the amount of phases and fineness of cement (difference in PSD) has a significant effect on the characteristics of the early period of concrete, but at a later age has less effect [22]. The aim of this study was to determine the exact effect of C3A and C3S changes on the technical characteristics of fresh and hardened concrete (concretes without additives). A Laboratory mill was used to stabilize clinker grinding and cement grading conditions. More accurate identification of this issue in the second phase (separate from this project), i.e. industrial testing and proper control to implement the necessary measures, will help us a lot

2. MATERIALS AND METHODS

In the first stage, during three months, the clinker produced was sampled weekly in two kilns of Tehran Cement Factory. Chemical analysis was performed on all clinker samples by the wet method so that from these 25 clinker samples taken, 11 samples were selected whose phase values were varied. A sample of gypsum was prepared, and a chemical analysis was performed. Laboratory cementation was performed under the same conditions using 11 clinker samples during the second phase. Laboratory cementation with a combination of 96% clinker and 4% gypsum with the criterion of achieving Blaine index was 300±5 m2/kg. A sample of super plasticizing additive (polycarboxylate) was also prepared. In the third phase, the physical and mechanical properties of the cement (determination of residue on sieves, determination of fineness with Blaine index, setting time, normal consistency, and compressive strength of standard mortar) of cement samples were measured (Figure 2).

Figure 2. Laboratory cementation and determination of their physical and mechanical characteristics

During the fourth phase, 22 concrete mixtures with 11 cement samples were prepared with the aim of achieving a slump of 8±0.5 cm, so that 11 mixtures with the title of control (without additives) and 11 mixtures with the title of additives. Figure 3 and Table 2 show the characteristics of aggregates used in this study.

Table 2. Technical specifications of materials used in concrete mix designs

Type	Passing percent of sieve 200	Fracture (%)	ρ (SSD)	Water absorption (%)	fineness modulus (FM)	MSA
gravel	0.1	79	2.53	2.64	*	**19**
sand	0.2	*	2.54	3.88	3.5	*

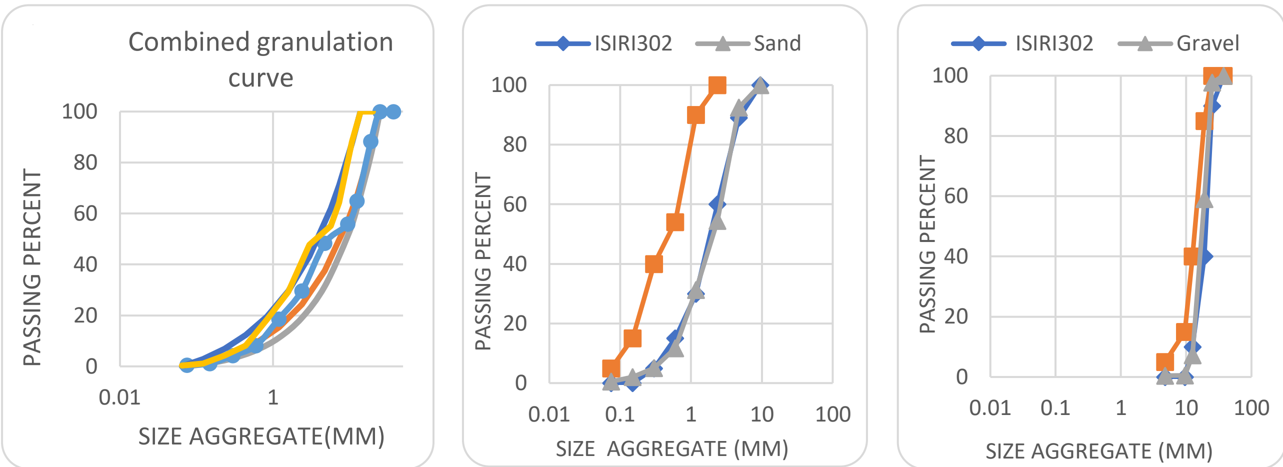


Figure 3. Graphs of sand (right), sand (middle) and composition of materials (left) used in concrete mixes

In all mixtures, the quality of aggregate and water materials, weight values of materials, physical conditions such as temperature, tools used, Examiner and processing conditions have been as constant as possible so that by creating the same conditions, only the clinker phases used in cement are variable (Figure 2). The grade of cement was 350 kg/m3, and 65% sand with 35% gravel was used. [23] (Figure 5). On concrete mixtures, fresh air conditioning, density, slump loss within one hour, and compressive strength of hardened concrete of 7, 28, and 90 days were performed. On the concrete mix with additives, tests were performed to determine the fresh concrete air density, determine the slump loss trend within one hour, determine the fresh concrete air loss trend within one hour, and compressive strength of hardened concrete for 7, 28, and 90 days. Intuitive characteristics of all fresh concrete mixtures were recorded for comparison (Figure 4).

Figure 4. Preparation of concrete mixes and tests

3. RESULTS AND DISCUSSION

Table 3 shows the chemical analysis of the clinker samples used in this study. According to the results, C3S values are about 48-61%, and C3A values are about 4-7%. Therefore, there are various clinkers for cementing.

Table 3. Chemical analysis of clinker samples used in laboratory cement

Sample Code	Free CaO	C4AF	C3A	C2S	C3S	ALM	SIM	LSF	CaO	Fe2O3	Al2O3	SiO2
11	0.70	14.0	6.0	25.8	49.6	1.13	2.25	89.7	63.5	4.6	5.2	**22.02**
7	1.01	12.8	5.0	26.5	50.0	1.09	2.56	89.1	63.12	4.2	4.56	**22.4**
2	0.84	11.7	4.5	21.6	56.3	1.08	2.79	91.2	63.72	3.84	4.16	**22.32**
3	0.39	12.1	3.7	16.5	61.4	0.99	2.77	93.2	63.88	3.96	3.94	**21.9**
10	0.48	13.6	5.8	28.0	48.1	1.13	2.35	88.8	63.54	4.48	5.04	**22.4**
4	0.70	12.5	6.1	35.7	41.5	1.19	2.58	85.8	63.34	4.12	4.92	**23.34**
6	0.56	11.9	6.1	25.1	51.2	1.23	2.54	90.0	63.36	3.92	4.82	**22.2**
5	0.39	11.9	6.0	25.8	51.3	1.22	2.60	89.8	63.86	3.9	4.76	**22.5**
1	0.42	12.3	7.4	23.5	51.9	1.33	2.32	91.0	63.84	4.04	5.38	**21.84**
8	0.45	11.8	5.6	19.4	57.8	1.19	2.59	92.4	64.16	3.88	4.6	**21.96**
9	0.42	12.2	5.7	26.3	50.4	1.18	2.57	89.4	63.48	4	4.72	**22.44**

Analysis of gypsum used for laboratory cementation was as shown in Table 4 This chemical analysis shows that the gypsum used is of good quality.

Table 4. Chemical analysis of gypsum used in laboratory cementation

Molecular water	CaSO4	SO3	MgO	CaO	Fe2O3	Al2O3	SiO2
18.42	70.6	41.54	0.78	31.8	0.22	0.78	1.84

Table 5 shows the results of physical and mechanical analysis of laboratory cements.

Table 5. Results of physical and mechanical analysis of laboratory cements

Code	Compressive Strength-Mortar (kg/cm2)			Setting Time (min)		Normal	Retained on sieve (%)		Blaine (cm2/gr)
	2 Days	**7 Days**	**28 Days**	**Initial**	**Final**	**Consistency (%)**	**90µ**	**45µ**	
1	180	316	472	230	310	23.5	1.34	12.04	2951
2	176	291	430	230	300	23.5	1.32	11.36	3052
3	172	303	343	240	315	24.0	1.30	11.46	3052
4	132	286	466	230	310	23.5	1.18	12.26	2951
5	206	335	492	215	290	24.0	1.78	13.64	2985
6	163	321	493	240	315	23.5	1.24	12.84	2951
7	146	286	466	215	295	23.5	1.34	15.42	2985
8	222	307	514	190	265	23.5	0.88	10.22	2985
9	167	308	494	240	315	23.5	1.24	12.34	3019
10	150	311	534	240	320	24.0	1.20	12.22	2951
11	169	296	446	250	325	23.5	2.08	14.20	3019

To check the setting time, Figure 5 shows the diagram of the aluminate amount and the initial and final setting time. Due to the fact that in this project the amount of gypsum used was the same for all cement factories (4%), the changes in setting time were only due to changes in phase C3A that this inverse relationship was not observed in the initial setting results. Extremely similarity of the results of normal concentration of samples (23.5-24%) can be effective on this issue.

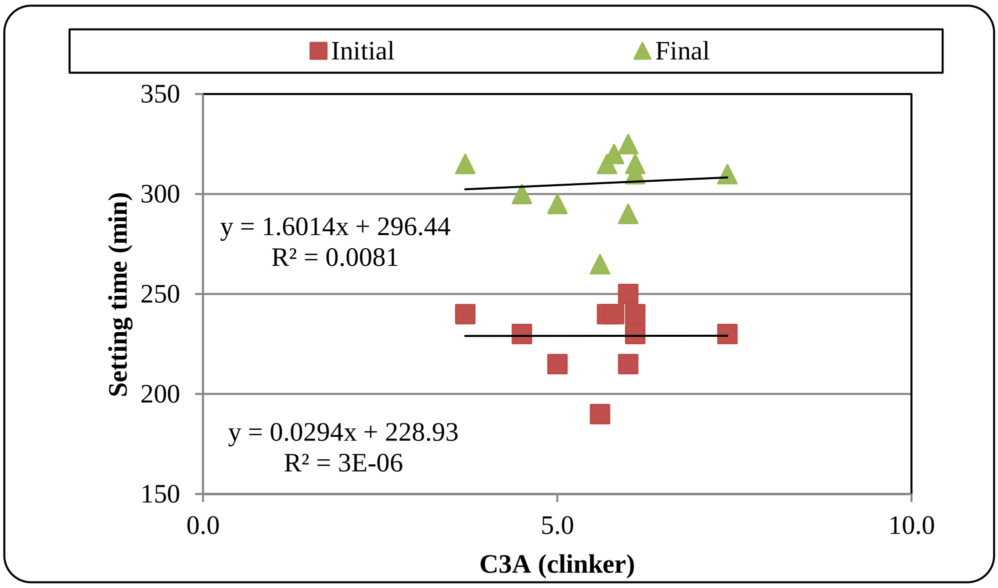


Figure 5. Diagram of the relationship between alumina values and initial and final setting time of laboratory cement samples

In the next step, the compressive strength results of concrete mixtures (Table 6) are investigated.

Table 6. Test results of concrete mixtures made with laboratory cement

Code	Without Admixture (Normal)					With Admixture				
	Fresh Concrete		Compressive Strength-Concrete (kg/cm2)			Fresh Concrete		Compressive Strength Concrete (kg/cm2)		
	Slump (mm)	w/c	7 Days	28 Days	90 Days	Slump (mm)	w/c	7 Days	28 Days	90 Days
1	80	0.55	247	397	488	80	0.47	250	411	479
2	90	0.54	248	392	427	85	0.48	286	458	438
3	80	0.59	219	377	433	80	0.54	236	373	468
4	80	0.56	209	392	499	80	0.51	225	389	479
5	80	0.53	246	440	545	80	0.48	272	429	438
6	80	0.57	239	382	490	80	0.52	254	401	425
7	80	0.54	278	439	582	80	0.50	272	336	564
8	80	0.54	284	428	451	80	0.48	328	443	505
9	80	0.51	274	453	496	80	0.46	299	462	463
10	85	0.56	251	366	500	85	0.51	277	399	464
11	80	0.54	271	390	470	80	0.50	272	363	463

Comparison of the effect of C3S clinker value on compressive strength of standard mortar and concrete showed that with increasing C3S value, compressive strength (mortar and concrete) increase (Figure 6). Given the correlation index of these results, a clear relationship cannot be imagined. In general, it can be said that with each percentage increase of C3S clinker, about 1.5 units (kg/cm2), the strength of 7 and 28 days of concrete increases. On the other hand, with the increase of C3S from about 57% onwards, the decrease of mortar and concrete strength is clearly seen, which can be pointed out to the negative effect of C3S phase overload.

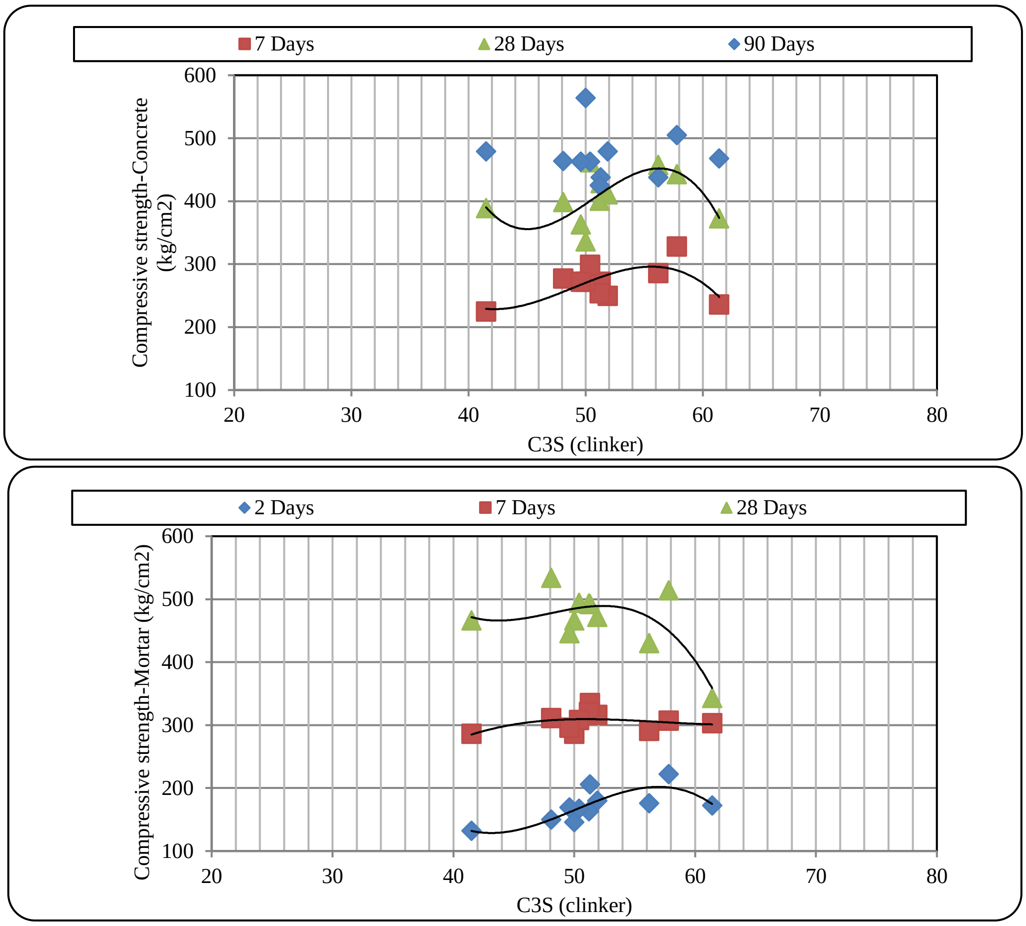


Figure 6. Diagram of the relationship between Alite values and initial and final setting time of laboratory cement samples

According to the references in the minor role of C3A in providing (short-term) strength of concrete, according to the comparison of the effect of C3A clinker on the compressive strength of standard mortar and concrete in this study, with increasing C3A (up to about 5%), compressive strength (mortar and Concrete) showed a clear increase (Figure 7). Due to the weak correlation of these results, a clear formula for this relationship cannot be imagined.

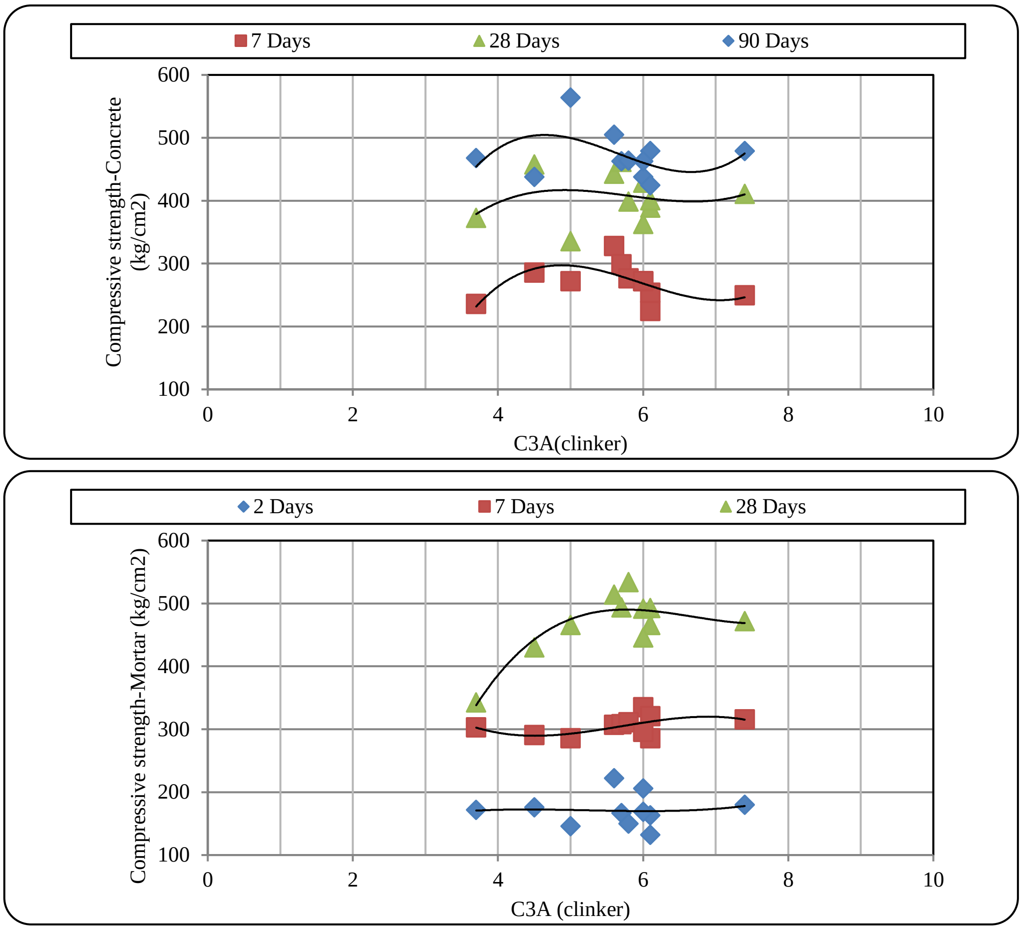


Figure 7. Diagram of the relationship between aluminate values and initial and final setting time of laboratory cement samples

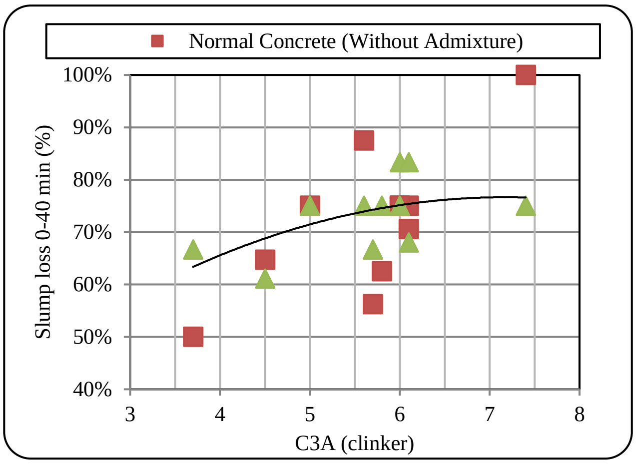
Table 7 shows the results of tests and observations of fresh concrete mixtures in this study. Based on the results in this table, it can be said that:

As C3A decreases, the properties of fresh concrete improve slightly, which may be related to the demand for more alumina cement (explanation that with increasing aluminate, the demand for cement increases, and for concrete producers who operate in the traditional way. And to achieve the desired performance and the desired slump, water is added to the concrete, increasing the ratio of water to cement and thus reducing the compressive strength of concrete,

- With the reduction of C3S, the properties of fresh concrete are slightly improved (the explanation of the previous paragraph also applies to this case,
- Consumption of concrete admixture in the concrete mix with admixture has shown a slightly negative effect on the characteristics (delay) of fresh concrete (e.g., slump loss trend) compared to control concrete mix (no additive).
- Comparison of the effect of C3S clinker value on fresh concrete indices showed that increasing the C3S value up to about 55% improves the slump loss of concrete, but with increasing this phase, negative effects on the two parameters are seen (Figure 8).
- This comparison for the C3A parameter showed that as the C3A increases, the slump decreases, which is not desirable (Figure 8).

Table 7. Test results of concrete mixtures made with laboratory cement

6	5	4	7	9	3	8	1	11	2	10	6	5	4	7	9	3	8	1	11	2	10	Clinker no.
With additive											control											Mix name
8.0	8.0	8.0	8.0	8.0	8.0	8.0	8.0	8.0	7.5	8.0	8.0	8.0	8.0	8.0	8.0	8.0	8.0	8.0	8.0	8.5	8.0	Initial slump (cm)
12	12	12	11.5	12	12	12	12	11.5	9	12	*	*	*	*	*	*	*	*	*	*	*	Slump after additive (cm)
2.18	2.28	2.40	2.23	2.30	2.23	2.28	2.30	2.30	2.20	2.33	2.28	2.35	2.28	2.28	2.40	2.30	2.38	2.23	2.25	2.28	2.33	Density (gr/cm3)
5	5	5	5	5	5	5	5	5	5	5	5	5	5	5	5	5	5	5	5	5	5	Consistency
5	5	5	5	5	5	5	5	5	5	5	5	5	5	5	5	5	5	5	5	5	5	Workability: Initial (5min)
5	4	4	4	5	5	5	5	5	5	5	5	5	5	5	5	5	5	5	5	5	5	Workability: Delayed (20min)
5	5	5	5	5	5	5	5	4	5	5	5	5	5	5	5	5	5	5	5	5	5	finishing: Initial (5min)
5	4	4	4	5	4	5	5	4	5	5	5	4	4	5	5	4	5	5	5	5	5	finishing: Delayed (20min)
5	5	5	5	5	5	4	4	5	5	5	5	5	5	5	5	5	5	5	5	5	5	Compaction (by hand)
5	5	5	5	5	5	5	5	4	5	5	5	5	5	5	5	5	5	4	5	5	5	Adhesion of concrete components together: Initial (5min)
5	5	5	5	5	5	5	5	4	5	5	5	5	5	5	5	5	5	5	5	5	5	Adhesion of concrete components together: Delayed (20min)
5	5	5	5	5	5	5	5	5	5	5	5	5	5	5	5	5	5	5	5	5	5	No rough texture
5	5	5	4	5	5	5	5	4	5	5	5	5	5	5	5	5	5	5	5	5	5	No separation
5	5	5	5	5	5	5	5	5	5	5	5	5	5	5	5	5	5	5	5	5	5	No long setting time of concrete
5	5	5	5	5	5	4	5	4	5	5	5	5	5	5	5	5	5	5	5	5	5	No bleeding (5)
5.00	4.83	4.83	4.75	5.00	4.92	4.83	4.92	4.50	5.00	5.00	4.87	4.88	4.89	4.91	4.91	4.91	4.92	4.92	4.97	4.97	5.00	Average point
12.0	12.0	12.0	12.0	12.0	12.0	12.0	12.5	12.0	9.0	12.0	8.0	8.0	8.0	8.0	8.0	8.5	8.0	8.0	8.0	8.5	8.5	slump (cm): 0 min
7.0	6.0	7.0	6.0	7.5	4.5	4.0	8.0	8.5	5.5	6.5	4.5	6.0	6.0	3.5	4.5	4.5	4.5	4.0	6.0	7.0	6.5	slump (cm): 20 min
3.0	3.0	4.0	3.0	3.0	2.0	2.0	4.0	4.5	3.5	3.0	2.0	3.0	3.5	1.0	2.0	2.5	2.0	2.0	4.0	3.0	0.0	slump (cm): 40 min
1.5	1.0	1.5	1.0	1.0	0.5	1.0	2.0	1.0	0.0	3.0	1.0	1.0	1.0	0.0	1.0	1.0	0.0	1.0	2.0	1.0	0.0	slump (cm): 60 min



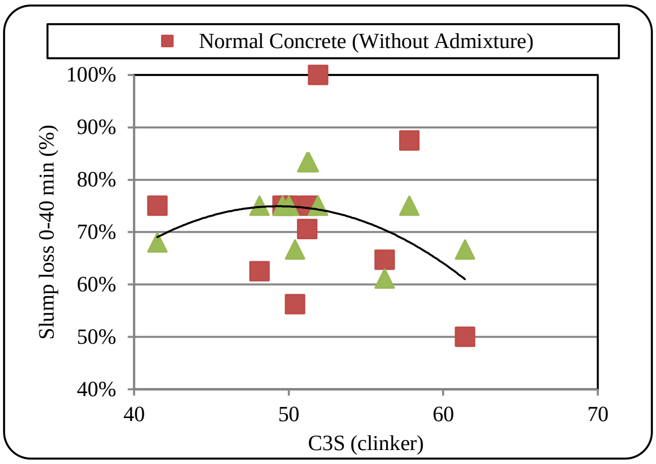


Figure 8. Diagram of the relationship between the values of Alite, Aluminate and slump loss of laboratory concrte mixtures

Comparison of the effect of C3A clinker on the amount of fresh concrete air showed that with increasing the amount of this phase, void almost increases. Additive application caused void of concrete, which did not show a clear trend.

Based on the results of air determination experiments of fresh concrete mixtures containing additives and its declining trend in this study, it was found that the optimal value of C3A is 6-5% (Figures 9).

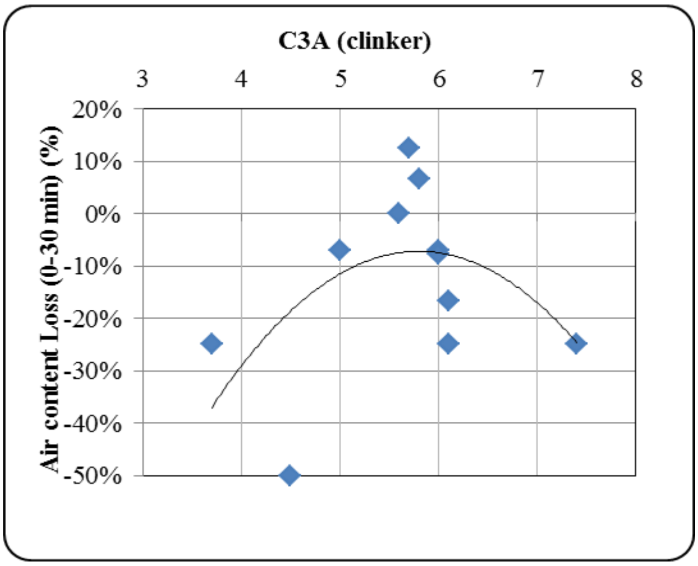


Figure 9. Diagram of the relationship between alumina content and concrete air loss for concrete mixtures containing additives

4. CONCLUSION

Based on the results of this project, the effect of the number of clinker phases on the technical characteristics of cement and concrete is as follows:

1- Grinding results (Blaine and residue on sieves) of laboratory cement indicated similar conditions to clinker wear, which makes comparability valid;

2- With increasing C3S, compressive strength (mortar and concrete) increases. According to the correlation index of these results, no clear relationship was seen. In general, it can be said that with each increase of one percent C3S clinker, about 1.5 units (kg/cm2), the strength of 7 and 28 days of concrete increases. On the other hand, with the increase of C3S from about 57% onwards, the decrease in mortar and concrete strength was clearly seen;

3-In concrete mixtures, with the increase of C3S to more than 55%, the ratio of water to cement also showed a complete increase, which caused a decrease in strength;

4- With increasing the amount of C3A (up to about 5%), the compressive strength (mortar and concrete) showed a clear increase;

5-Additive consumption had a small negative effect on the properties of fresh concrete;

6-With the reduction of C3A, the properties of fresh concrete improved slightly;

7- With increasing the amount of C3S up to about 55%, the trend of slump loss and concrete density improved;

8. With an increasing amount of C3A phase, void almost increases. The results of air determination tests of fresh concrete mixtures containing additives and its declining trend in this study showed that the optimal value of C3A is 5-6%.

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AUTHORS CONTRIBUTION

This work was carried out in collaboration among all authors.

CONFLICT OF INTEREST

The author (s) declared no potential conflicts of interests with respect to the authorship and/or publication of this paper.

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