

**SELF-CONSOLIDATING TERNARY SYSTEMS WITH
LOW SHRINKAGE CAPACITY**

**DÜŞÜK RÖTRE KAPASİTELİ KENDİLİĞİNDEN
YERLEŞEBİLEN ÜÇLÜ SİSTEMLER**

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ABSTRACT

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Self-compacting cementitious systems are gaining popularity in several areas of construction such as pile foundations, repair works and floor finishings since their easy application and feature to minimize the application errors make them preferable. Ternary system consisting of Portland cement, calcium aluminate cement and any calcium sulfate has also desirable features originated from the properties of each ingredient, it contains. Its properties can be modified according to the relative amount of binders with each other. By adjusting the rates, it is possible to provide rapid setting, high early strength, and shrinkage compensation to the composite. In addition, types of the binders play a significant role to control the behavior of the composite.

The self-compacting ternary systems which combine the two mentioned systems' properties above, make ground in construction sector, by offering many head starts. With this composite, it is possible to cover extensive areas in a very short time with an ultimate

high strength easily. In addition, to provide high volume stability to the composite is possible.

This thesis aims to develop ternary systems that can be brought into service in short periods after application and be used in floor finishings, repair, retrofitting, and connecting the precast structures, considering the cost of the composites. For these purposes, Portland cement is replaced with more economic calcite powder and superplasticizer is used as an additive to the system by 0.5 % and 2.5 % by weight of Portland cement and calcium aluminate cement to obtain a self-compacting system. The self-compacting system's setting time is arranged using set retarders while shrinkage-reducing admixture and polypropylene fibers are used to reduce the shrinkage rate of the system. As a result, a self-compacting ternary system with a 48.8% better shrinkage rate than the ultimate shrinkage rate given in ASTM-C-596 is obtained.

Keywords: ternary system, self-compacting cementitious system, low shrinkage capacity, self-compacting ternary systems

ÖZET

DÜŞÜK RÖTRE KAPASİTELİ KENDİLİĞİNDEN YERLEŞEBİLEN ÜÇLÜ SİSTEMLER

Utku BELENDİR

Yüksek Lisans, İnşaat Mühendisliği Bölümü

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Kendiliğinden yerleşen çimento esaslı sistemler, kolay uygulanmaları ve uygulama hatalarını en aza indirme özellikleri nedeniyle kazık temeller, onarım işleri ve zemin kaplamaları gibi inşaatın çeşitli alanlarında popülerlik kazanmaktadır. Portland çimentosu, kalsiyum alüminat çimentosu ve herhangi bir kalsiyum sülfattan oluşan üçlü sistem, içerdiği her bir bileşenin özelliklerinden kaynaklanan arzu edilen özelliklere de sahiptir. Özellikleri, bağlayıcıların birbirleriyle olan göreceli miktarına göre değiştirilebilir. Oranları ayarlayarak kompozite hızlı priz, yüksek erken dayanım ve rötre dengelemesi sağlamak mümkündür. Buna ek olarak, bağlayıcı türleri kompozitin davranışını kontrol etmede önemli bir rol oynamaktadır.

Yukarıda bahsedilen iki sistemin özelliklerini bir araya getiren kendiliğinden yerleşen üçlü sistemler, birçok avantaj sunarak inşaat sektöründe çığır açmaktadır. Bu kompozit ile çok kısa sürede çok geniş alanları kolaylıkla nihai yüksek mukavemet ile kaplamak mümkündür. Ayrıca kompozite yüksek hacimsel stabilite sağlamak da mümkündür.

Bu tez çalışmasında, kompozitlerin maliyeti göz önünde bulundurularak, uygulamadan kısa bir süre sonra hizmete alınabilecek ve zemin kaplamalarında, onarım, güçlendirme ve prekast yapıların birleştirilmesinde kullanılacak üçlü sistemlerin geliştirilmesi amaçlanmıştır. Bu amaçlar doğrultusunda Portland çimentosu yerine daha ekonomik olan kalsit tozu ve süperakışkanlaştırıcı sisteme katkı maddesi olarak Portland çimentosu ve kalsiyum alüminat çimentosunun ağırlıkça %0,5 ve %2,5 oranında kullanılarak kendiliğinden yerleşen bir sistem elde edilmiştir. Kendiliğinden yerleşen sistemin priz süresi priz geciktiriciler kullanılarak düzenlenirken, sistemin rötre oranını azaltmak için rötre azaltıcı katkı ve polipropilen lifler kullanılmıştır. Sonuç olarak, ASTM-C-596'da verilen nihai büzülme oranından %48,8 daha iyi büzülme oranına sahip kendiliğinden yerleşen üçlü bir sistem elde edilmiştir.

Anahtar Kelimeler: Üçlü sistem, kendiliğinden yerleşebilen çimentolu sistem, düşük rötre kapasitesi, kendiliğinden yerleşebilen üçlü sistem

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SYMBOLS AND ABBREVIATIONS

Abbreviations

C	Calcite
CA	Citric acid
CAC	Calcium aluminate cement
Cs	Calcium sulfate
PC	Portland cement
PPF	Polypropylene fiber
RS	Rochelle Salt (Sodium potassium tartrate)
SCM	Self-compacting mortar
SP	Superplasticizer
$\mu\epsilon$	Micro strain
σ	Flexural strength

1. INTRODUCTION

Self-compacting cementitious system is a cementitious system that flows itself due to gravity without needing compaction or vibrations. The self-compacting cementitious system applications have been used for several benefits. Such as self-compacting systems have faster and easier application than the traditional methods, provide smooth and aesthetic finishes, and reduce labor costs. These systems are mostly used in retrofitting and repair, floor finishings, drilled shafts, etc. However, for having the self-compacting ability the mixture has limited solid content and higher binder ratios than other cementitious systems which may cause volumetric instability due to shrinkage which eventually causes cracks.

In recent years, ternary systems consisting of calcium aluminate cements - Portland cements - calcium sulfate sources (PC-CAC-Cs), have been used for high early strength, and lower shrinkage capacity. These features make ternary systems suitable to use in repair and high surface area applications such as floor finishing. The main purpose of using the ternary system is to eliminate the disadvantages of PC and CAC, such as the C_3A phase forms unstable forms in cementitious strength that cause strength reduction in later ages but in the presence of Cs, the calcium aluminate cement produces more stable phases. For PC, its shrinkage rates are higher than the CAC and the early strength is low but with CAC higher early strengths can be arranged.

The advantages of self-compacting systems and ternary systems can be combined to be used for floor finishings, repair, retrofitting, and connecting the precast structures. However, the knowledge about self-compacting ternary systems is limited in the literature. So, in this thesis, self-compacting ternary systems with low shrinkage capacity are investigated step by step and it was aimed the final mixture to have at least 25 cm flow diameter, 2.5 MPa flexural strength and 15.0 MPa compressive strength on 3rd day, 6.0 MPa flexural strength and 30.0 MPa compressive strength on 28th day with lower shrinkage capacity than the ultimate shrinkage value from ASTM-C 596 and the self-compacting reference sample containing Portland cement as the main binder.

Chapter 1 contains a general information about the composite with ternary system and the scope of this study. A literature review about the subject is given in Chapter 2. The materials, processes, and methodology used in the study are described in detail along with the methodology in section 3. The discussions of results are given in the following chapter, Chapter 4. The study is finalized with the findings of the thesis and recommendations in Chapter 5.

2. LITERATURE REVIEW

2.1. Self-consolidating Cementitious Systems

Self-consolidating mortar refers to a type of mortar that has the ability to flow under its own weight without external interference. The feature of this mortar increases the productivity of screed applications from 1500 m²/day to 2400 m²/day [1]. Such mixes must have adequate segregation resistance and be readily deformable to attain self-compactability. Ideal viscosity and low yield stress are necessary for the fresh matrix to have good deformability and avoid phase separation [2].

The self-compacting feature is generally measured with three types of tests which are the mini slump test, flow rate test, and self-placing test. For the mini slump test, a cone is used without consolidation sequence by filling the mixture to cone then the cone is lifted after that the sample collapses so that the maximum the spreading diameter mix was determined to calculate the deformability index or compare the mean values of diameters. In the flow rate test, self-compacting material is poured into a funnel, and the material can flow out freely after the bottom stopper is removed. The drain down time of the material is measured. After that, the flow rate index is calculated by the formulation $\frac{10}{t}$ where “t” is the measured time. For the self-placing test, a box vessel with two chambers divided by a barrier is used. In the partition surface, three reinforcing bars are placed with 1.5 cm distance from each other. One of the chambers is filled with self-compacting concrete then the partition is removed. The height reached by in-flow is measured. From measured height the degree of self-leveling is measured by the formulation: $\frac{h_i}{(h_0/2)}$ where h_i is measured height, h_0 is the height of the materials in the first chamber before removing partition [2,3].

In the research done by Kong et al. [2], a self-compacting cementitious composite (ECC) by improving the parameters, including fresh state and rheological properties was developed. To obtain self-compacting ECC, ordinary Portland cement and silica sand whose particle sizes range from 0.2 to 0.3 mm, and some chemicals, formaldehyde sulfate (SP) and hydroxypropyl methylcellulose (HPCMC) were used. SP was added to the matrix after other ingredients to prevent false setting and fibers were added at last manually to the matrix. In the experiment, the w/c ratio of the specimens was 0.3 where the content of SP was 4 %. Also, the fiber content was 1 %, by volume while the fiber length was 12,5 mm so the specimens have high deformability to show self-compacting concrete. The specimens showed the self-compactability in fresh state.

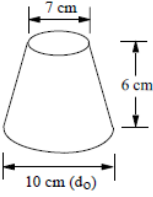
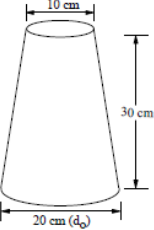
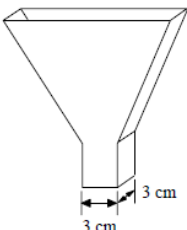
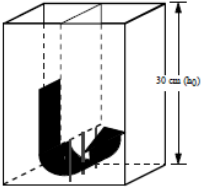
Test	Deformability test I (Flow table test)	Deformability test II (Slump flow test)	Flow rate test (Funnel test)	Self-placing test (Box vessel test)
Dimensions of test device	Flow cone with 10 cm in bottom diameter and 6 cm in height 	Slump cone with 20 cm in bottom diameter and 30 cm in height 	Funnel with a 3 × 3 cm outlet and cm ³ in total volume 	2-chamber vessel with max. height of 30 cm and 12 mm-rebars in 2.5 cm of spacing at the connection 
Type of measurement	Diameter of the spread, \bar{d}_2 (cm)	Diameter of the spread, \bar{d}_1 (cm)	Time for material to vacate from the outlet, \bar{t} (sec.)	Height reached by the inflow material, \bar{h} (cm)
Corresponding index	Deformability, F_1	Deformability, F_2	Relative flow rate, R	Degree of self-leveling, L
Conversion	$\frac{\bar{d}_2 - \bar{d}_0}{\bar{d}_0}$	$\frac{\bar{d}_1 - \bar{d}_0}{\bar{d}_0}$	$\frac{10}{\bar{t}}$	$\frac{\bar{h}}{(h_0/2)}$
Note	Standard test for fresh mortar except no consolidation is applied	Standard test for quality control of fresh concrete	Dimensions of funnel proposed by Ozawa et al for mortar test	Dimensions scaled down for ECC

Figure 1 Deformability tests held by Li et al. [2]

Zeng et al. [4] investigated the effect of 3 different water-reducing admixtures on the properties of anhydrate-based floor screeds with self-leveling property. As water-reducing admixture (WRA) polycarboxylate superplasticizers, sulfonated naphthalene-formaldehyde condensate (SNF)- and sulfonated melamine formaldehyde (SMF)- based materials were

used. In the research, the water/binder was 0.38 and the amounts of the chemical admixture were determined using the overall amount of binders. The results showed that polycarboxylate-based material was the most effective one compared to others. There was a significant impact on $\text{CaSO}_4 \cdot \text{H}_2\text{O}$ generation, which led to a decrease in mechanical properties and an increase in shrinkage. The coagulation and hardening characteristics of the mixes were similar for all types of WRAs as well as the distribution of air pore sizes in self-leveling mixtures containing PCE, SMF, and SNF. Nonetheless, PCE may increase the potential of micropore association by affecting the hydration mechanism coarsening the pore diameter, which reduces mechanical features.

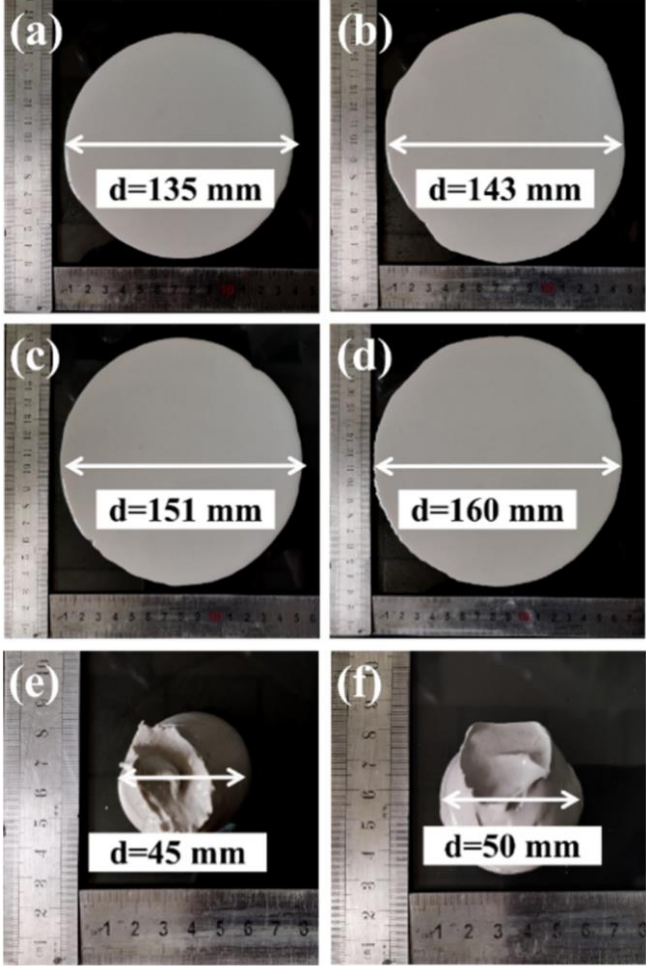


Figure 2 Flowability tests done by Zang et al. [4], (a) 0.12 % polycarboxylate SP; (b) 0.15 % polycarboxylate SP; (c) 0.18 % polycarboxylate SP (d) 0.21 % polycarboxylate SP; (e) 0.21 % SMF; and (f) 0.21 % SNF.

Akhmetov et al. [5] investigated the feasibility and necessity of using fibers to study the deformative properties of self-compacting concrete (SCC) to improve its technical and physical parameters. To achieve the improvement by using fibers, polypropylene fibers (PPFs) having lengths of 6,9,12,15 mm in quantity from 0.5 to 2 kg/m³ of the SCC mixture were used. The fiber usage higher than 2 kg/m³ led to a rapid decrease in flow values which could not provide self-compacting ability to all mixtures. Longer fiber lengths improved the properties better than the short ones and using the fiber having the length of 15 mm with 1kg/m³ content increased flexural strength by 10,5 % compared to the sample with no fiber content. The higher fiber content and longer fiber sizes decreased the drying shrinkage in the research as in Figure 3.

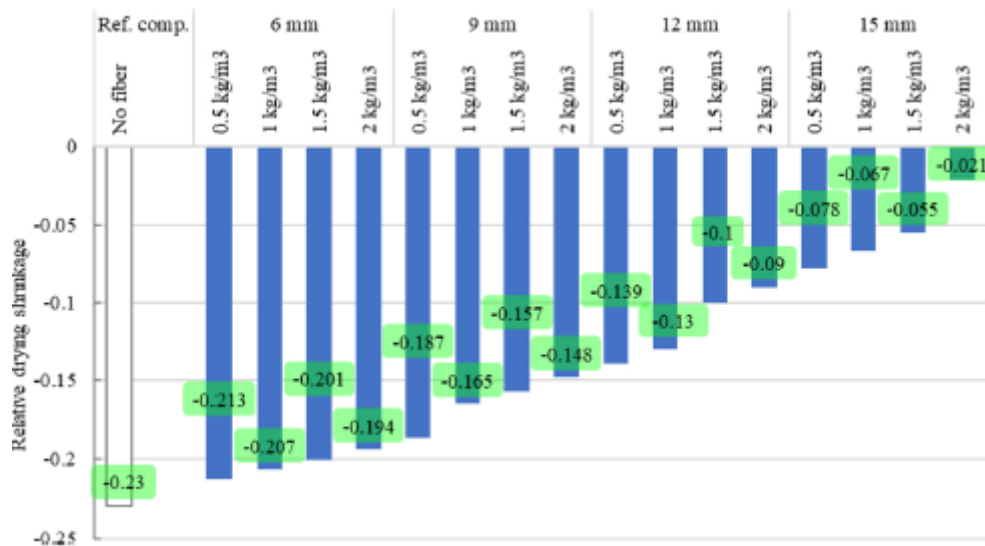


Figure 3 The drying shrinkage results from the study of Akhmetov et al.[5].

The effects of using limestone fines as a substitution for cement and sand on self-compacting concrete's (SCC) properties were examined both in its fresh and hardened states. by Skender et al. [6]. The different replacement amounts for sand and ordinary Portland cement were tested and it was found that increasing the amount of limestone fines decreased the slump flow for both replacements. In this experiment, the mechanical properties decreased, gradually with increasing limestone replacement of cement content, but when substituted for sand, it resulted in an increase in compressive strength.

Şahmaran et al. [3] examined the performance of different chemical and mineral admixtures in self-compacting mortar (SCM). To achieve this, 3 SPs, 2 viscosity modifying admixtures, limestone powder, fly ash, brick powder, and kaolinite were used as mineral additives. The mineral replacement amounts were 15 % and 30 % of the cement weight. From the experiment, SP was the main parameter that affected the consistency and modified polycarboxylate-based SP showed better performance for workability than polycarboxylic ether and melamine formaldehyde. Limestone and fly ash improved the workability however kaolinite and brick powder decreased it so they could not be used as a single additive but could be used in combination with other mineral additives. The chemical and mineral admixtures increased setting time while fly ash increased the most. The mineral additives used in the study decreased the strength compared to the chemical admixtures when they were used as cement replacements.

Rani and Priyanka [7] studied to determine the maximum amount of polypropylene fibers in self-compacting concrete. Fly ash was used as a cement replacement and the fiber was used as an additive to the concrete with ratios of 0 %, 0.25 %, 0.50 %, 0.75 %, 1 %, and 1.25 % by weight in the experiment. From the results, 1 % usage of fiber increased the flexural strength the most while the best compressive strength was achieved from 0.75 % fiber ratio. After 1 % fiber usage, the strength decreased, and it was concluded that the use of fibers up to 1 % was feasible in manufacturing SCC.

Demir et al. [8] investigated the effects of adding shrinkage-reducing admixtures (SRA) to self-compacting concrete (SCC). The SRA usage were between 0.8 % to 1.2 % of the cement by weight. The SRA additive in given dosages had a slight impact on fresh properties and reduces the surface tension of concrete. The additive of SRA reduced the shrinkage especially for early ages. SRA usage not only delayed the cracking age and decreased the propagation of the cracks. SRA addition decreased the flexural and compressive strengths of the mixtures but the reduction was relatively insignificant.

2.2 Ternary Systems

The ternary systems, which are composed of ordinary PC-CAC-Cs, contribute to cementitious systems having lower shrinkage and high early strength. When the CAC

content in the system is higher the system tends to have rapid setting, and high early strength but tends to have lower compressive strength. The ratio of these 3 components can be arranged according to needs. In Figure 4 PC rich ternary system is highlighted.

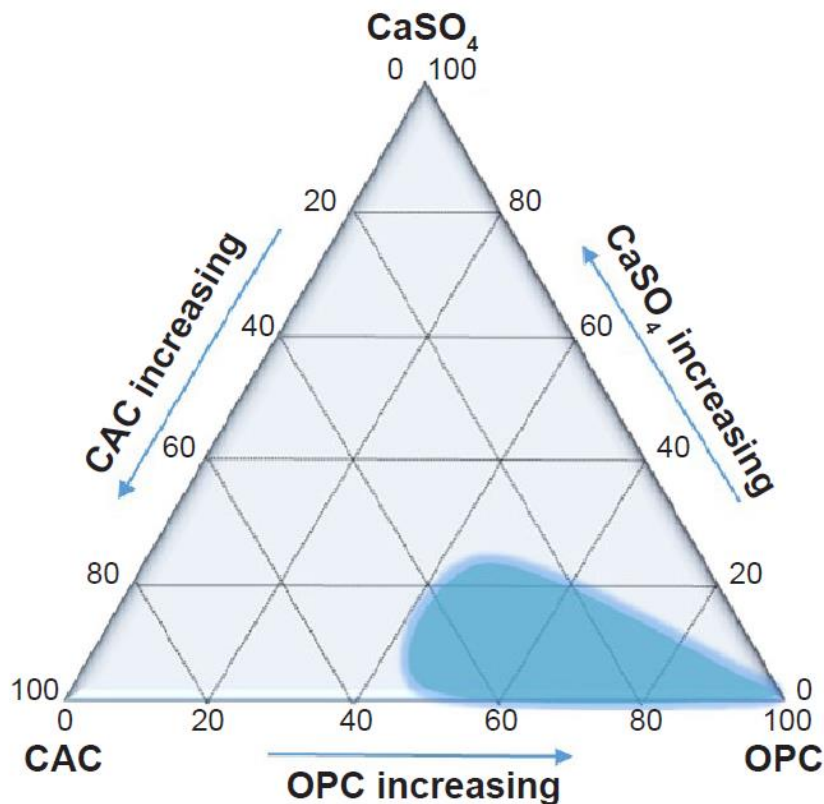


Figure 4 OPC-CAC-Cs ternary system, highlighted area shows PC-rich systems [1]

In a different study [9] the impact of component dosage on the expansive cement's volume change and hardening processes, as well as on the hydration kinetics and the type and amount of hydrates that form, was examined. Gypsum (96.5 % $\text{CaSO}_4 \cdot 2\text{H}_2\text{O}$) and anhydrite (95.58 % CaSO_4) were utilized as sources of calcium sulfate. The expansive admixture/PC ratios were from 15/85 to 25/75 while the calcium sulfate/CAC ratios varied between 0.55 and 0.80. The study showed that:

- In the experiment the main hydrates formed in the PC-CAC- gypsum/anhydrite system were: ettringite, portlandite, C-S-H, and solid solutions ($3\text{CaO} \cdot \text{Al}_2\text{O}_3 \cdot \text{CaSO}_4 \cdot 12\text{H}_2\text{O}$ - $4\text{CaO} \cdot \text{Al}_2\text{O}_3 \cdot 13\text{H}_2\text{O}$).
- The ettringite formation proportion was lower in the compositions with anhydrite than those containing gypsum.

- The maximum ettringite formed after 1 to 3 days for gypsum-containing specimens, and 3 to 7 days for specimens including anhydrite.
- For early ages, because of an increase in the calcium sulfate rate, the hydration process was slowed down at early ages (first day). and this effect was more important for gypsum-containing compositions than anhydrite and altered the rate at which ettringite forms; at extended hydration times, an increase in the CaSO₄/CAC ratio increased the formation of larger ettringite crystals.
- Higher content of PC generally increased the compressive strength and after one to three days of hydration, the strength values decrease with the increase of the content of calcium sulfate.

Table 1 The chemical and mineralogical compositions of OPC, CAC (40 %, 50 %, 60 %, 70 %, 80 % alumina content), and C₁₂A₇-rich clinker [1]

Binder material	Chemical composition				Mineralogical composition	
	Al ₂ O ₃	CaO	Fe ₂ O ₃	SiO ₂	Main phases	Secondary phases
OPC	-	++++	-	+	C ₃ S, C ₂ S	C ₃ A, C ₄ AF, C
40% alumina	++	++	+	-	CA, C ₄ AF	C ₁₂ A ₇ , C ₂ S
50% alumina	+++	++	-	-	CA	C ₂ AS
60% alumina	+++	++	-	-	CA	CA ₂ , C ₂ AS
70% alumina	++++	+	-	-	CA, CA ₂	αA
80% alumina	++++	+	-	-	CA, CA ₂ , αA	-
C ₁₂ A ₇ rich clinker	++	+++	-	-	C ₁₂ A ₇ , C ₄ AF	C ₂ S, CA

+ indicates intensity in dosage; - present, but not significant.

Bizzozero et al. [10] examined the mechanism of expansion in Cs-containing calcium aluminate and calcium sulphoaluminate (CSA) systems. In this experiment systems containing both CAC and CSA cement that have had gypsum added, exhibit unstable expansion above a specified level (approximately 45 mol % of calcium sulfate for CAC systems and approximately 55 mol % for CSA systems). The amount of expansion at 28 days gradually increased below this threshold. On one hand below that threshold, samples stayed intact for at least 65 days, and at the beginning initial swelling was observed then the samples' dimensions remained stable. On the other hand, the samples above this threshold continued to expand gradually until fail. Regarding the overall amount of ettringite, the distribution of size of pores, and the microstructures, the systems did not show any noticeable distinctions. Two mechanisms work together to explain the expansion, which is

a threshold effect. First, the range of pore sizes where ettringite could be formed and the amount of stress that can be applied was determined by its supersaturation. It had to do with how well ettringite grows within a network of interconnected pores; beyond a critical sulfate level, a higher percentage of porosity in the ettringite may caused the overall volume of ettringite to increase, which would create pressure and trigger the start of the unstable expansion. Only a limited number of isolated pores were able to bear pressure from ettringite below the critical sulfate threshold. Second, confined ettringite formation was putting pressure on the system. This stress surpassed the elastic limit in high-expansion systems, and cracking caused unstable expansion and failure.

Son et al. [11] investigated the effect of CaSO_4 on hydration and phase conversion of CAC. In the experiment, CaSO_4 ratios were selected as 2,4,8 % of the CAC where the water/CAC ratio was 0.4 by weight. The compressive strength reduction, porosity, and pore sizes decreased with the CaSO_4 ratio increased. Increasing ratios of CaSO_4 content increased the ettringite formation which hindered the development of the metastable phase CAH_{10} , which is in charge of the strength decline in later ages. Monosulfate formation caused CaSO_4 to trigger pore-size refinement.

Benabed et al. [12] studied the rheological and mechanical features of self-compacting mortars (SCMs) with different types of sand (crushed, river, and dune), and the combination of these sand types. In the experiment, limestone fines were used at varying percentages up to 30 % and SP dosage was changed to achieve the slump of 280 ± 20 mm for each mixture. According to the test results when the limestone content increased the mini slump flow decreased. However, up to 15 % of limestone fines increased the compressive strength. Due to the dune sands' fineness, the mixture's water demand increased, and the samples showed lower flowability compared to the others. While the mixtures created with binary and ternary sand remained within SCM limits for slump flow and V-funnel time, their compressive strength increased significantly. It was concluded that the usage of two or three sands combined in SCC applications has economic and environmental benefits production.

Wang et al. [13] studied the different effects of CACs on hydration processes of the PC-blended systems. For that purpose, 2 CACs with different monocalcium aluminate content

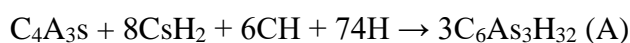
were used in varying amounts. The CAC amount which started from 3 % CAC content to 15 % with 3% increments was chosen. It was found that the CAC content below 6 % had minor effect on the initial and final setting time whereas setting time reduced above 6 % and rapid setting occurs at 15 % CAC content. The compressive strength of the mixtures decreased by increasing the CAC amount and higher content of monocalcium aluminate in CAC results in lower compressive strength. Moreover, it was concluded that higher CAC content and monocalcium aluminate delayed the hydration of PC.

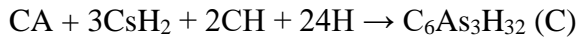
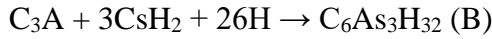
Qoku et al. [14] examined the effects of the sulfate source on the evolution of the solid phase assemblage and hydration in ternary PC, CAC, and Cs systems. The hydration mechanism of the investigated mixes of PC-rich and CAC-Cs-rich samples were similar. Most of the hydration was influenced by the kind of sulfate source. When compared to the gypsum pastes, the anhydrate-bearing samples produced more ettringite. Furthermore, for the CAC-Cs-rich formulations, higher amounts of stratlingite were found in the presence of gypsum as opposed to anhydrate, indicating the effect of the source of sulfate on the phases that carry silicates' hydration.

2.3. Self-consolidating Ternary Systems

Self-consolidating ternary systems have some advantages like higher early strength and better volume stability than the cementitious self-consolidating systems. Self-consolidating systems have limited solid volume and high cementitious content in the matrix compared to cementitious systems, which can lead to shrinkage. Expansive binders and additives that reduce shrinkage are used to prevent such scenarios. The ternary systems consisting of CAC, Cs, and ordinary OPC are one of these expansive binder types.

Dimensional stability is achieved with the application of a compensatory mechanism through the formation of the ettringite ($\text{CaO} \cdot \text{Al}_2\text{O}_3 \cdot 3\text{CaSO}_4 \cdot 32\text{H}_2\text{O}$) for ternary systems. The hydration reactions of the hydraulic binders which are rich in CaO, Al_2O_3 , and SO_3 must be used to control its formation. Some reactions with different cement types that form ettringite are shown in the equations, the equation A is Portland cement blended with anhydrous calcium sulphaaluminate, gypsum and free lime, the equation B is PC with high amount of tricalcium aluminate and gypsum and the equation C is PC blended with calcium aluminate and gypsum.





where C=CaO, A=Al₂O₃, s=SO₃, H=H₂O, C₄A₃s=4CaO.3Al₂O₃. SO₃ - anhydrous calcium sulphoaluminate (CSA, ye'elinite), CsH₂=CaSO₄.2H₂O - gypsum or dihydrate, CH=Ca(OH)₂ - calcium hydroxide, CA=CaAl₂O₄ - calcium aluminate, and C₃A=Ca₃Al₂O₆ - tricalcium aluminate C₆As₃H₃₂=ettringite [1].

Xu et al. [15] examined how different curing temperatures affected the hydration of ternary binders dominated by CAC. In the experiment, the ternary binder mass ratios (PC:CAC: calcium sulfate) were kept constant at 22.5:51.7:25.8 for all specimens with water to binder ratio of 0.5. With this research, they reached the following conclusions:

- The determined ternary binders' primary hydration products were primarily associated with the phases based on calcium sulphoaluminate. Ettringite to plate-like monosulfate converted sooner at greater temperatures, particularly for the binders doped with anhydrite.
- In the pastes with anhydrite, the temperature increased to 40 °C promoted the formation of stratlingite and Al(OH)₃. After three days, pastes prepared with anhydrite had a coarser pore size distribution; pastes containing hemihydrate, on the other hand, clearly produced finer pores. The hemihydrate-doped system's pores primarily ranged between 100 - 300 nm after three days of storage at 10 °C. When the temperature rose to 40 °C, the majority of the pores are found in the range of 10 nm to 50 nm.
- Hemihydrate was better for the applications in high-temperature conditions, whereas, at lower curing temperatures, anhydrite-based mortars can only achieve a desirable one-day strength for PC:CAC:Cs of 22.5:51.7:25.8.

In another research, the ternary systems with PC/CAC/Cs compared to the binary systems composed with PC/CAC, showed as better volume stability as shown in Figure 5. [1]

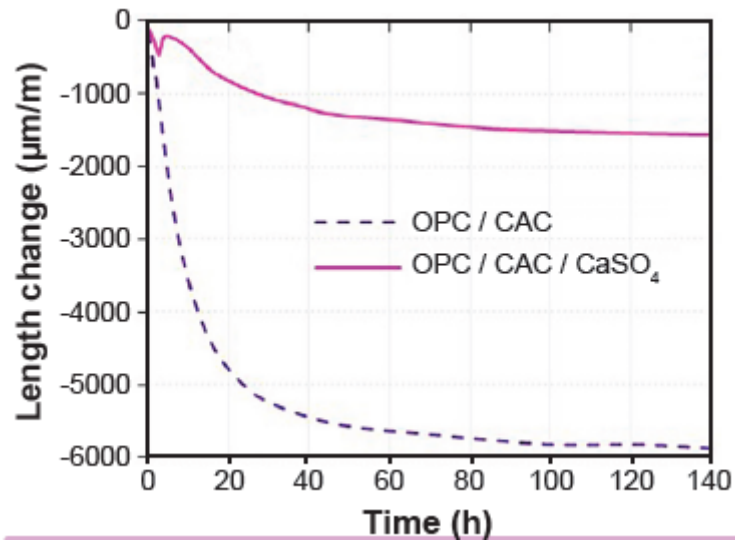


Figure 5 Length change of ternary system and binary system in the research given in Rengo et al. (2021) [1]

The ratio of the ternary systems significantly impacts the properties of the mixture including length change over time. According to the research done by Zhang et al. [16], the Cs type and content in the mixture changed the shrinkage amount. In Figure 2.5 3 different plaster types (α -hemihydrate, anhydrite, and dihydrate) with amounts of 0 %, 2 %, 4 %, and 6 % by mass are shown. The increase in the Cs dosage increased the flexural and compressive strength until the maximum value, then decreased. Dihydrate type of Cs had more early strength while the rapid rate of dissolution led to a higher expansion of α -hemihydrate. Self-leveling mortar prepared with the anhydrite type of Cs showed the lowest fluidity loss and had more setting time than the ones containing other types.

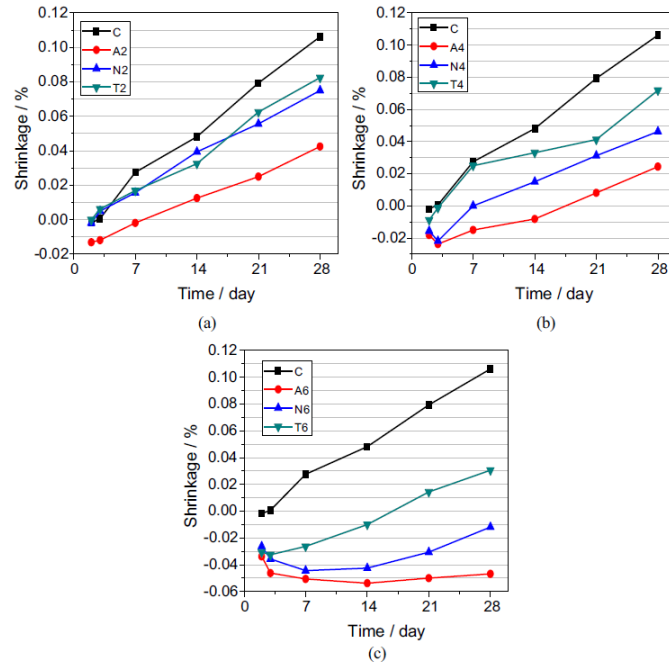


Figure 6 Drying shrinkage results from the research of Zhang et al. (2018) (a. 2 % Cs; b. 4 % Cs; c. 6 % Cs) [16]

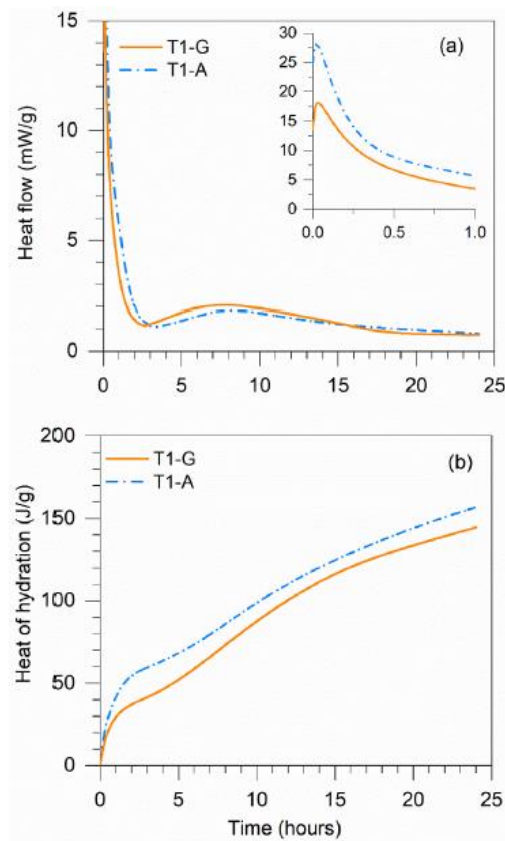


Figure 7 Heat of hydration for PC rich (PC:CAC:Cs =80:13:7) mixtures where T1-G contains gypsum and T1-A anhydrite as Cs source [14]

3. MATERIALS AND METHODOLOGY

3.1. Materials

The materials used in this study are given under this title. The ternary system is used as a binder which consists of Portland cement (PC), calcium aluminate cement (CAC), and calcium sulfate (Cs).

3.1.1. Cements

In this study, CEM-I 42.5 R commercially obtained from LİMAK Cement and the second one was Votorantim Cimentos brand CEM-II /A-LL 42.5 R Portland cements were used. Chemical composition of the CEM-II type cements is given in Table 2. Both cements are suitable for TS EN 197-1.

Table 2 Composition of CEM-II / A-LL 42.5 R type cement.

Ingredient	Content (%)
CaO	62.97
SiO ₂	20.46
Al ₂ O ₃	5.94
SO ₃	3.66
Fe ₂ O ₃	3.40
MgO	1.87
K ₂ O	0.70
Na ₂ O	<0.02
Loss of Ignition	0.20



Figure 8 Digital image of a) CEM-I cement b) CEM-II cement.

ISIDAÇ40 was used as calcium aluminate cement (CAC) obtained from ÇİMSA. The chemical composition of CAC is given in table 3.

Table 3 Chemical composition of CAC.

Ingredient	Composition (%)
SiO ₂	3.93
Al ₂ O ₃	39.81
Fe ₂ O ₃	17.64
CaO	35.71
MgO	0.76
K ₂ O	0.16
Na ₂ O	0.08
SO ₃	0.01
P ₂ O ₅	0.06
TiO ₂	1.92
Cr ₂ O ₃	0.0746
Mn ₂ O ₃	0.0968



Figure 9 Digital image of ISIDAÇ40.

3.1.2. Calcium Sulfate

Alpha-type hemihydrate calcium sulfate from the BALDUDAK brand was used as a Cs source in this study. The technical data of the Cs source is given in Table 4.

Table 4 The technical data of Cs.

Type	α
Consistence (plaster/water gr/gr)	2
Flow diameter (mm)	190±20
Freeze start (min.)	10±3.5
Freeze end (min.)	Max 30
Flexural strength (MPa)	7.85
Compressive strength (MPa)	18.63
Hardness (shore D)	75
Linear expansion (%)	0.36
Water absorption (%)	25



Figure 10 Digital image of alpha-type calcium sulfate hemihydrate

3.1.3. Sand

Quartz sand was used in the study with a maximum aggregate size of 1 mm. The calcite has 400 μm maximum aggregate size and its chemical composition is given in Table 3.4. The digital images of the sand and calcite are given in Figure 5.

Table 5 Chemical composition of calcite.

Symbol	Concentration (%)
SiO ₂	0.0800
Al ₂ O ₃	0.17
Fe ₂ O ₃	<0.0010
CaO	49.34
MgO	5.333
SO ₃	0.040
Na ₂ O	0.2176
K ₂ O	0.33
TiO ₂	<0.0015
Cr ₂ O ₃	0.00028
MnO	<0.00099
P ₂ O ₅	0.0322
ZnO	<0.00037
SrO	0.01220
Loss of ignition	44.4500

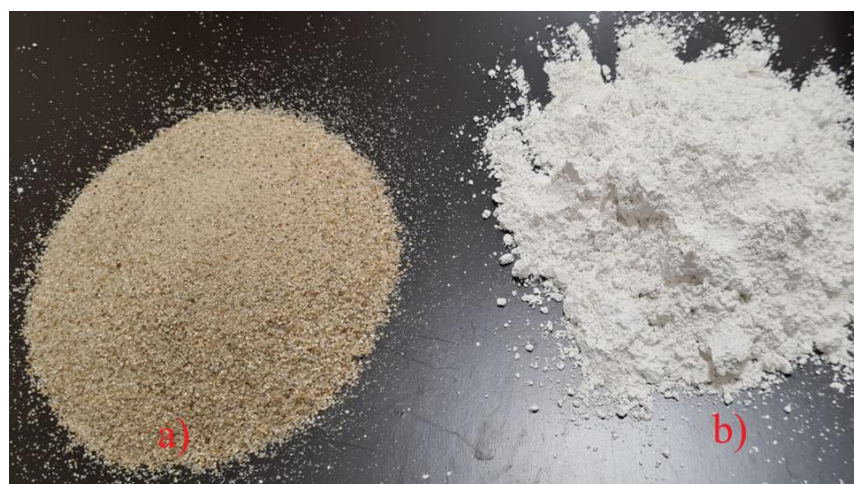


Figure 11 Digital images of a) sand, b) calcite.

3.1.4 Admixtures

3.1.4.1. Shrinkage Reducing Admixture

In the experiment, the SRA-10 obtained from PERAMIN company was used (Figure 12). The admixture is in powder-form with 65-71 % active substance. Its specific bulk density is about 0.35.



Figure 12 Digital image of PERAMIN SRA 10.

3.1.4.2. Superplasticizer

A polycarboxylate-based superplasticizer (SP) CEMESTE P 97-H and a naphthalene-based (sodium naphthalene sulfonate formaldehyde) EBA 1446 SP were used in the study.



Figure 13 Digital image of the superplasticizers used in the study a) polycarboxylate-based CEMESTE P 97-H b) naphthalene-based SP

3.1.4.3. Set Retarders

Two types of set retarders were used in the study. The first one was citric acid ($C_6H_8O_7$) (CA) and the second one was sodium potassium tartrate (Rochelle salt, $KNaC_4H_4O_6 \cdot 4H_2O$) (RS). The properties of the set retarders are given in Table 6.

Table 6 Properties of the set retarders.

Property	Sodium potassium tartrate	Citric acid
Appearance	Powder, crystalline powder, crystalloid	Powder, crystalline powder, crystalloid
Color	Colorless, white	Colorless, white
Solubility	-	Water soluble
Melting Point ($^{\circ}C$)	70-80	135-152
Degradation Temperature ($^{\circ}C$)	-	345
Boiling point ($^{\circ}C$)	399.3	-
Density (g/cm ³)	1.79	1.542-1.665

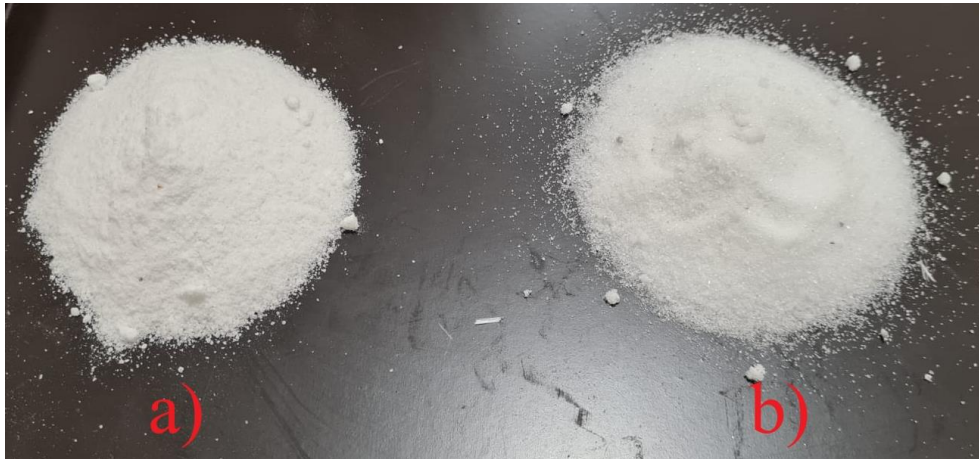


Figure 14 Digital image of the set retarders a) $\text{KNaC}_4\text{H}_4\text{O}_6 \cdot 4\text{H}_2\text{O}$ (Rochelle salt) b) $\text{C}_6\text{H}_8\text{O}_7$ (citric acid)

3.1.5 Fibers

In the study, polypropylene fibers (PPF) (Figure 15) obtained from ATA KİMYA were used. The features of the PPF are given in Table 7.



Figure 15 Digital image of polypropylene fibers.

Table 7 Features of PPF.

Purity	100 % polypropylene
Type	Homopolymer
Color	Natural white
Cross section	Circular
Standard	ASTM-C1116
Fiber length (mm)	6
Tensile strength (MPa)	350
Density (g/cm ³)	0.91
Point of softening (°C)	140
Point of melting (°C)	165-170
Acid resistance	Resistive
Oxidation resistance	Good
Organic solvent effect	Resistive
Biological resistance	Resistive
Cement suitable	Suitable
Alkali effect	Resistive
UV resistance	Resistive
Abrasion resistance	Good
Water absorption	Zero

3.2 Methodology

All procedures and test methods including casting, setting time analysis, mini slump test, shrinkage measurements, and mechanical tests are covered in-depth in this section.

3.2.1. Mixture proportions

The mixtures were prepared step by step to develop a self-compacting ternary system with low shrinkage capacity. In every part, the most suitable formulation was chosen for the main mixture used in the following step. The methodology followed is summarized in Figure 16. For the first step of the study, the binder ratios in ternary system were investigated and the composite yielding the highest flexural strength result was chosen as the main composite used in the following step. The ratio of water to binder was selected as 0.55 in this part to mold the samples more easily. Also, the 40 % of binder content was replaced with calcite since it was seen in literature, that calcite replacement may increase the workability [17] and calcite usage makes the mixture more economical. For the second step of the study, the water/binder ratio is reduced to 0.45 to have higher performance and volume stability [18]. In this step, the SP type and dosage were chosen by the flow diameter from mini slump test, and it is expected to exceed at least 25 cm [19]. The higher flow diameter without segregation is a better formulation to be used. In the third step of the study, the mixtures' initial setting time was adjusted to have longer workable time on site, by using set retarders. Then, the composite giving the lowest shrinkage result was tried to obtain by using SRA and PPF addition [5]. The mixture with the best shrinkage performance was chosen to be the mixture for optimization. The naming of the samples was done as shown in Figure 17. For each step, the addition type and amount was modified with respect to the performance, the new-added materials were shown in naming after the name of main composite. The initial T represented the main binder system used in composite, which is Ternary system. The first and second numbers represented the amounts of CAC with respect to total CAC and PC contents and Cs percentage with respect to CAC content, respectively. After them, the letter and number stood for the type of SP (C was for Cemeste, E was for EBA) and the percentage of SP with respect to total amount of CAC and PC content, respectively. The next numbers showed the percentages of CA and RS with respect to total CAC and PC content. The last numbers represented SRA percentage with respect to total CAC and PC content and PPF percentage with respect to total weight.

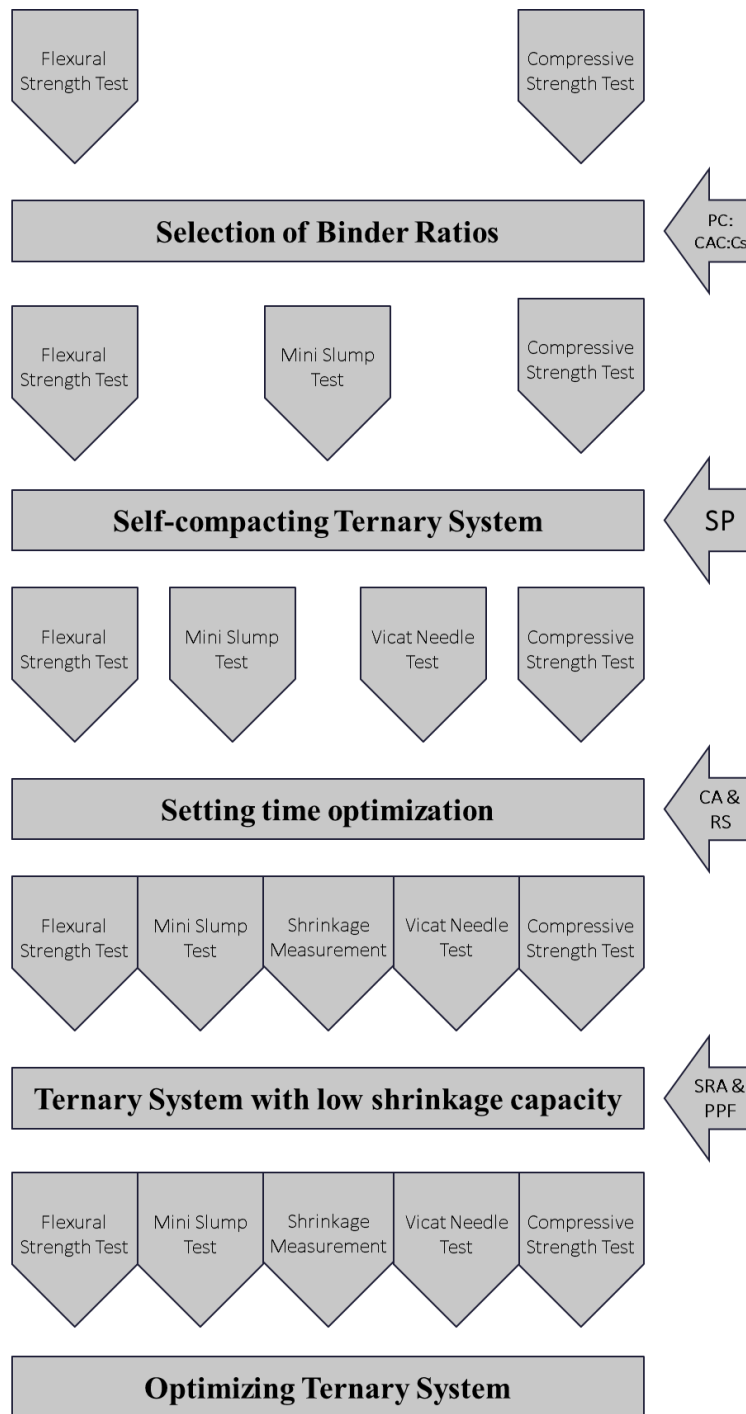


Figure 16 Schematic showing the procedure of the study.

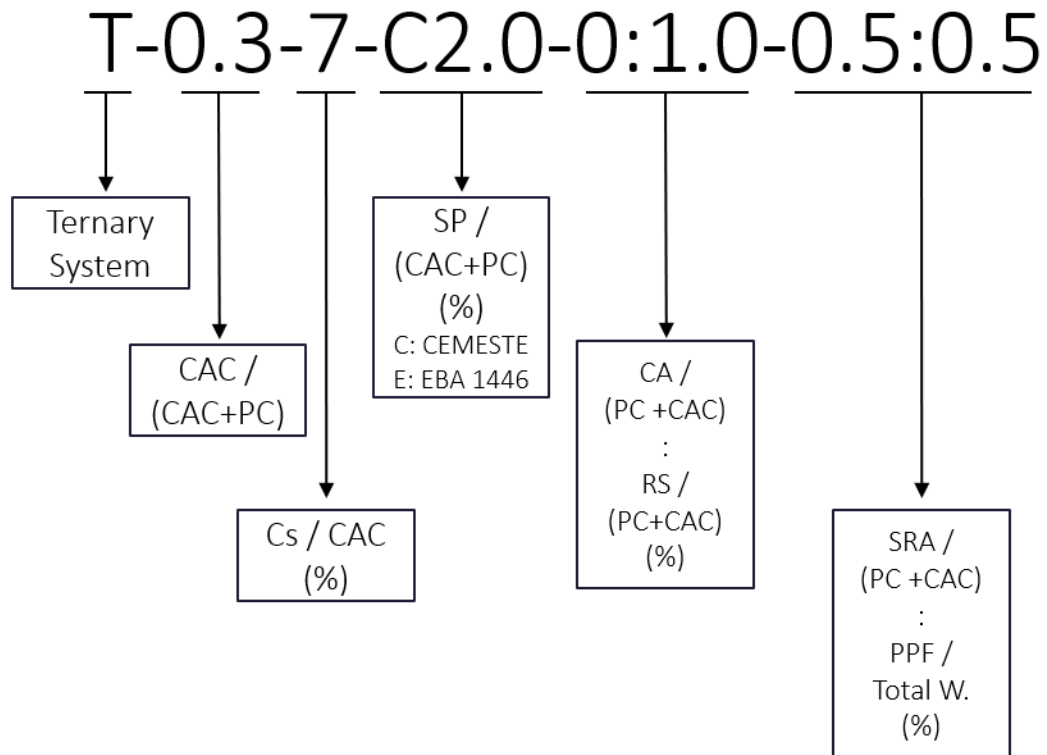


Figure 17 Naming of the samples.

3.2.2. Procedure

All powder materials including chemical admixtures and fibers, if any, were blended with a mixer (Figure 18) for 90 seconds to obtain a homogeneous mix, then water was added in additional 15 seconds at the slow rate. The total mixing time was adjusted to be 180 seconds.



Figure 18 Mixer used in the study.

For the first step of the study, the samples were put in two equal layers. For each layer, the composite was moved to the vibrating table to remove air voids completely as much as possible for 30 seconds (Figure 19). This method was used for the samples that are not self-compacting mixes. However, for all the following steps, the samples were directly poured into the molds without applying any compaction or vibrating process since they were expected to have self-compacting ability (Figure 20).



Figure 19 Samples placed to formworks by using the vibrating table.



Figure 20 Pouring self-compacting mixtures in formworks.

3.2.3. Testing

3.2.3.1. Mini Slump Test

To measure the mixture's self-compacting ability, a mini slump test was applied according to the procedure stated in EFNARC [19]. A thin oil layer was applied to a flat plywood which did not inhibit the movement of the sample and the metal ring with an upper diameter of 70 mm and bottom diameter of 100 mm with a height of 50 mm was placed into the middle. The sample was moved to the ring, then the ring was pulled up directly and after 10 seconds the maximum diameter and the diameter which is perpendicular to the max. diameter was measured and the average of the diameters was accepted as the flow diameter (Figure 21). It was assumed that the mix had a self-compacting ability if the flow diameter was higher than 250 mm without showing any segregation [19]



Figure 21 Mini slump test.

3.2.3.2. Setting Time Test

CAC has a rapid setting property, so the Cs source was used for arranging setting time in ternary systems [20]. To measure the initial setting time vicat needle (Figure 22) was used according to the TS EN 196-3 [21] but the measurement was done every 5 minutes due to the shorter initial times compared to the OPC-based systems. For the initial setting time, the

needle was put in a position in which touched the surface of sample for 2 seconds then it was allowed to penetrate from its position to the sample due to its own weight by releasing its rod. This action was repeated until the distance between the needle's initial location and the last location in sample was 4 ± 1 mm or the time duration needed for the movement was longer than 30 seconds. The time was recorded as the initial setting time.



Figure 22 Setting time test via Vicat needle.

3.2.3.3. Flexural Strength

The flexural strength is important for floor finishing since they have a very small thickness compared to their surface area. In this study, the procedure stated in TS EN 196-1 [22] standard was followed to measure the flexural strength (Figure 23). 4x4x16 cm samples were prepared and cured in ambient conditions until the test days when the testing days. The strengths of the composites were determined in 3rd, 7th and 28th days. A 3-point flexural

strength test was applied to the specimens with a 50 N/s loading rate and the support distance (L) is 100 mm. The flexural strength was calculated with the given formula:

$$\sigma = \frac{3 \cdot F \cdot L}{2 \cdot b \cdot d^2}$$

Where σ is flexural strength in MPa, F is force in N, L is the support length in mm, and b and d are sample width and height in mm, respectively.

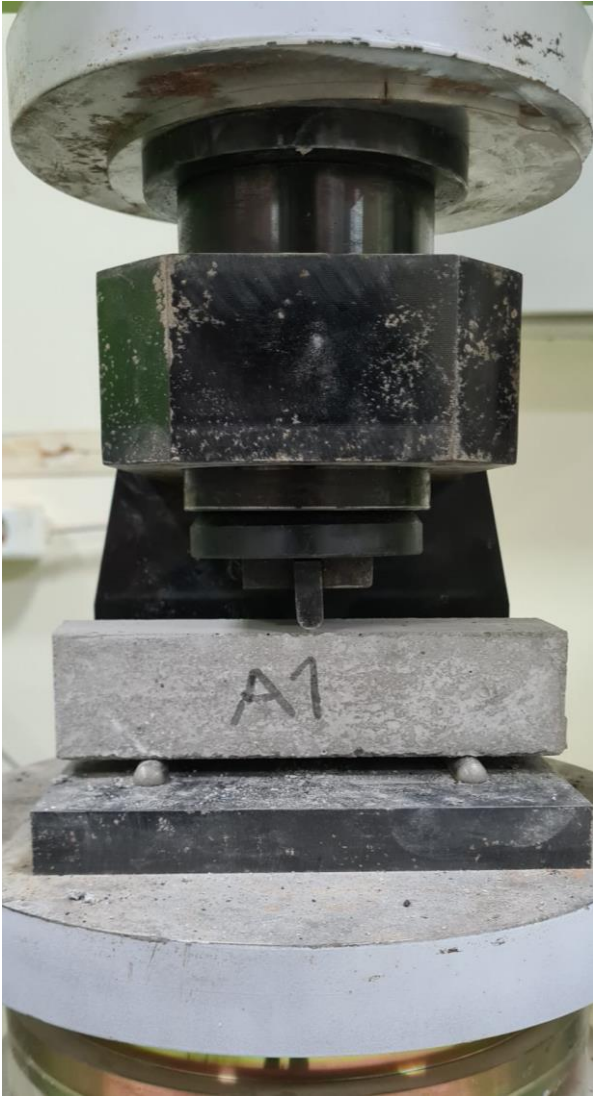


Figure 23 Flexural strength test.

3.2.3.4. Compressive Strength

The samples' compressive strengths were determined according to TS EN 196-1 [22] in 3rd, 7th and 28th days using the samples taken from the flexural strength test (Figure 24). The loading rate was set at 2.4 kN/s and the average result for two samples was considered as the compressive strength.



Figure 24 Compressive strength test.

3.2.3.5. Shrinkage

For the shrinkage measurement, the samples having the dimensions of 25 mm x 25 mm x 285 mm samples were prepared and cured in ambient conditions. The samples were demolded after 3 days from casting and initial measurements were taken afterward (Figure 25). The measurements were made according to ASTM C 596 [23], but the specimens were cured in ambient conditions to demonstrate the field conditions.



Figure 25 Drying shrinkage test.

4. RESULTS AND DISCUSSION

In this chapter, the results of the study are given. It was aimed that the final mixture to have at least 2.5 MPa flexural strength and 15.0 MPa compressive strength on 3rd day, 6.0 MPa flexural strength, and 30.0 MPa compressive strength on the 28th day with lower shrinkage capacity than the ultimate shrinkage value from ASTM-C 596 and the self-compacting cementitious system reference sample. To achieve that goal, the ternary binder ratios were determined in the first part. After that two types of SP were added to the chosen ternary system and suitable SP content was investigated to obtain a self-compacting ternary system. In the following step, to have an adequate working time, set retarders were added to the system. Then, shrinkage reducing materials, which are SRA and PPF were tried to reduce the shrinkage. The best mixture, that satisfied the scope of the study and lower shrinkage ratio was chosen to optimize. For the last part, the self-compacting ternary system was optimized by modifying the amount of set retarder and SRA.

4.1. Selection of Suitable Binder Content

4.1.1. Selection of CAC Content in Ternary System

In this section, the binder ratios in the ternary systems are investigated. For economic and environmental purposes, commercially obtained CEM II A/LL cement was used as the Portland cement in the system. Firstly, the CAC/(PC+CAC) ratio was investigated while the Cs/CAC ratio was kept at 0.07. The Cs content for the first step was decided according to the studies in the literature [9,11]. To increase the workability and volumetric stability, the 40 % of the binder is replaced by calcite powder (C) [17]. The sand (S) to the binder ratio was decided to be 1.25 [16] to provide the self-compacting ability and smooth surface to the composite. The water (W) to the binder ratio was chosen as 0.55 to mold the samples easily since it was seen that the lower water content in the ternary systems negatively affected the consistency of the mixtures in the preliminary studies.

The CAC/(PC+CAC) ratios were chosen as 0.1, 0.2, 0.3, and 0.4 to decide the optimum CAC content. The Portland cement mixture with the same amount of sand and water was also prepared for comparison purposes. The mixture proportions are given in the Table 8.

Table 8 The mix. designs of the cement mortars for the selection of CAC content in the ternary system.

Composite	PC / (PC + CAC)	CAC / (PC+CAC)	Cs / CAC (%)	C / (Binder + C)	S / Binder	W / Binder
PC	1.0	0.0	0.0	0.4	1.25	0.55
T-0.1-7	0.9	0.1	7.0			
T-0.2-7	0.8	0.2	7.0			
T-0.3-7	0.7	0.3	7.0			
T-0.4-7	0.6	0.4	7.0			

The flexural and compressive strength results of the mortars prepared with the cements are shown in Figures 26 and 27, respectively.

The increasing CAC/(PC+CAC) ratio up to 0.3, increased the flexural strength by 63.6 %, 74 %, and 50 %, but it was determined that it led to a decrease for 0.4 ratio by 36 %, 38 %, and 33 % at 3rd, 7th and 28th days, respectively. On the 28th day, the sample T-0.3-7 had 9.6 MPa flexural strength while PC had 6.4 MPa.

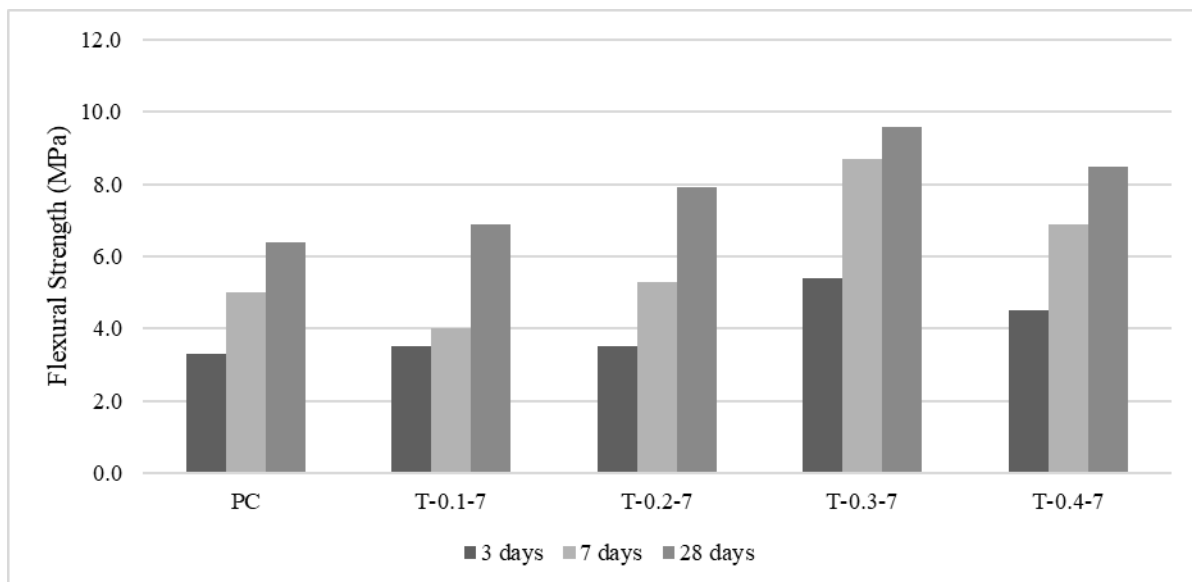


Figure 26 Flexural strength results for selecting the CAC content in the ternary system.

For the compressive strength results, a similar trend to the results of flexural strength was observed. At early ages, the ternary systems showed higher strength as expected due to the

CAC content. The highest result for 3rd day belonged to the sample T-0.2-7 with 28.1 MPa. For 7th and 28th days, the samples T-0.3-7 and T-0.4-7 showed the highest values with 36.6 MPa and 37.5 MPa, respectively. However, generally, it was observed that the compressive strength results for the samples T-0.3-7 and T-0.4-7 were comparable for all test ages.

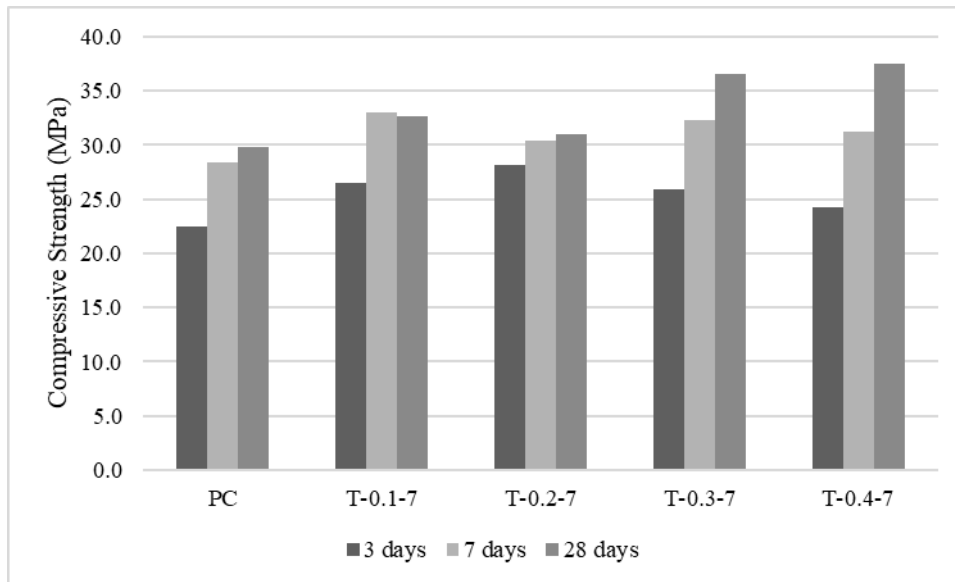


Figure 27 Compressive strength results for selecting the CAC content in the ternary system.

Since the composites having high flexural strength were preferred [18] for floor finishing and pavements and the highest flexural strength belonged to the sample T-0.3-7, the ratio of CAC content was decided to be 0.3 concerning the total content of CAC and PC.

4.1.2 Selection of Calcium Sulfate Amount in Ternary System

The effect of Cs content on both compressive and flexural strength of the ternary systems was investigated in this section. In this direction, three different Cs ratios to the total content of CAC were selected as 0.05, 0.07, and 0.09. The CAC/(PC+CAC) ratio was kept constant at 0.3 as determined in Section 4.1.1. The contents of other ingredients were adjusted as shown in Table 9.

Table 9 The mixture designs for the selection of Cs content in ternary system ratio

Composite	CAC / (PC + CAC)	Cs / CAC (%)	C / (Binder + C)	S / Binder	W / Binder
T-0.3-5	0.3	5.0	0.4	1.25	0.55
T-0.3-7	0.3	7.0			
T-0.3-9	0.3	9.0			

The flexural and compressive strength results of the composites are shown in Figure 28 and 29, respectively.

It was observed that when the Cs/CAC ratio increased from 0.05 to 0.07, the flexural strength increased from 4.4 MPa to 5.4 MPa for 3 days and from 6.0 MPa to 8.7 MPa for 7 days. At 28 days, the samples T-0.3-5 and T-0.3-7’s flexural strength results are comparable, the difference between them was just 3 %. However, above the ratio of 0.07, the flexural strength seemed to show a downward trend. The sample T-0.3-9 showed similar flexural strength performance to the sample T-0.3-5 for all ages except at the 28th day. The flexural strength results were 9.9 MPa, 9.6 MPa, and 8.8 MPa for the Cs/CAC ratios of 0.05, 0.07, and 0.09, respectively for 28th day.

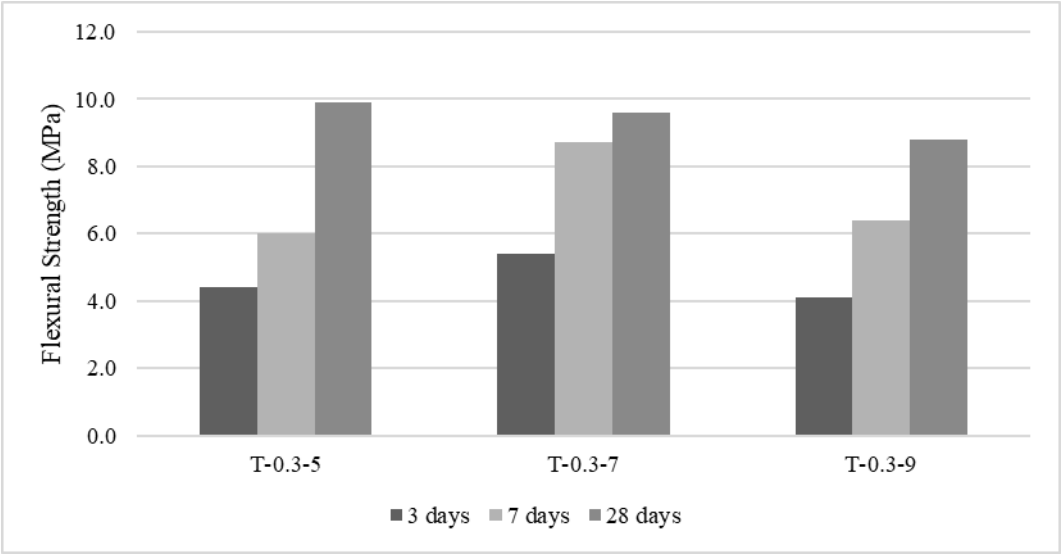


Figure 28 Flexural strength results for selecting the Cs content in the ternary system.

For the composites' compressive strength, the increase in Cs content in the mix led to the increase in early age strength. At later ages, the compressive strength results of all mixes were comparable. The samples T-0.3-5, T-0.3-7, and T-0.3-9 showed 39.9 MPa, 36.6 MPa, and 40.0 MPa compressive strength on the 28th day, respectively.

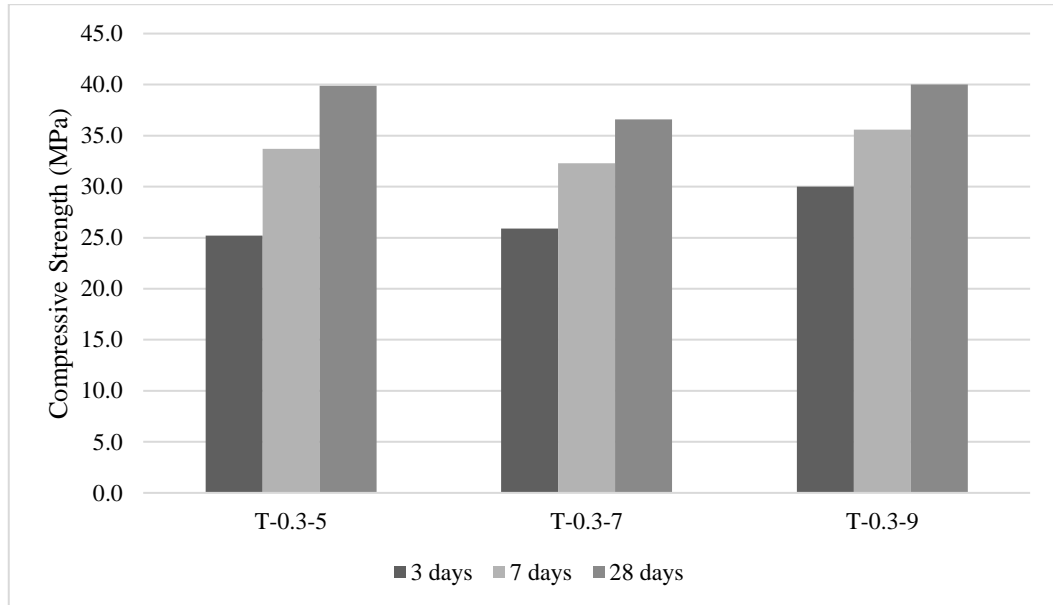


Figure 29 Compressive strength results for selecting the Cs content in the ternary system.

The ternary systems with different Cs contents had high flexural and compressive strength for all test days. But it should be noted that for ternary systems, the ratio of the flexural strength to the compressive strength was higher when they were compared to the normal Portland cement systems. For instance, the flexural and compressive strengths of the PC in Section 4.1.1, were 5.0 MPa and 28.4 MPa, for the 7th day, respectively (The ratio was $5/28.4=0.17$). But, for the sample T-0.3-7, these results were 8.7 MPa and 32.3 MPa (The ratio was $8.7/32.3=0.27$). It could be concluded that the flexural strengths of the ternary systems were relatively higher than those of the cementitious systems containing only Portland cement. A similar compressive and flexural strength correlation was also observed in the study of Zhang et al. [16]. The exact reason for that cannot be determined in the literature research, but the answer could lie in the microstructure of the hydration products of ternary systems, especially in ettringite.

The Cs content was chosen 7 % with respect to the content of CAC since this composite had generally higher flexural and compressive strengths than the samples T-0.3-5 and T-0.3-9.

4.2. Selection of Superplasticizer Type and Content

4.2.1 Selection of Superplasticizer Type

To select the suitable superplasticizer (SP), the performances of two common plasticizers (naphthalene-based [E] and polycarboxylate-based [C]) were compared in terms of flowability without segregation and strength of the composites for the same amount of SP [1]. The base mixture for this section was the sample T-0.3-7 (The contents of CAC and Cs were determined in Sections 4.1.1 and 4.1.2, respectively). The SP contents for both were chosen 2 % with respect to the total content of PC and CAC [2,3] The water/binder ratio was decreased to 0.45 to have no segregation in the fresh state of the composites. In addition, a decrease in the amount of water provided the reduction of shrinkage of the samples is expected [18]. The mix designs of the composites are given in Table 10.

Table 10 The mixture ratios for selecting the SP type.

Composite	CAC / (PC+C AC)	Cs / CAC (%)	C / (Binder + C)	S / Binder	SP(C) / (PC+CAC) (%)	SP(E) / (PC+CAC) (%)	W / Binder
T-0.3-7	0.30	7.00	0.40	1.25	0.00	0.00	0.45
T-0.3-7-C2.0					2.00	0.00	
T-0.3-7-E2.0					0.00	2.00	

After the mixing process, the mini-slump test was conducted. The flow diameters were 33.8 and 26.0 cm for the samples T-0.3-7-C2.0 and T-0.3-7-E2.0, respectively. The composite having polycarboxylate-based SP showed higher self-compacting ability than that having naphthalene-based SP. Also, the flow diameter of the E-included composite was at the minimum limits of the self-compacting value (25 cm) [19].

The flexural strength results are given in Figure 30. Regardless of its type, the use of SP as 2 % of the total content of PC and CAC decreased the flexural strength. The decreases for

the composite having SP(C) were 14.0 %, 24.0 %, and 6.9 % for the 3rd, 7th. and 28th days, respectively while the reductions in flexural strength for the SP(E)-incorporated one were 30.5 %, 52.8 % and 26.7 % for the same ages. The reason for that was the retarder effect of SPs used in cementitious systems [24].

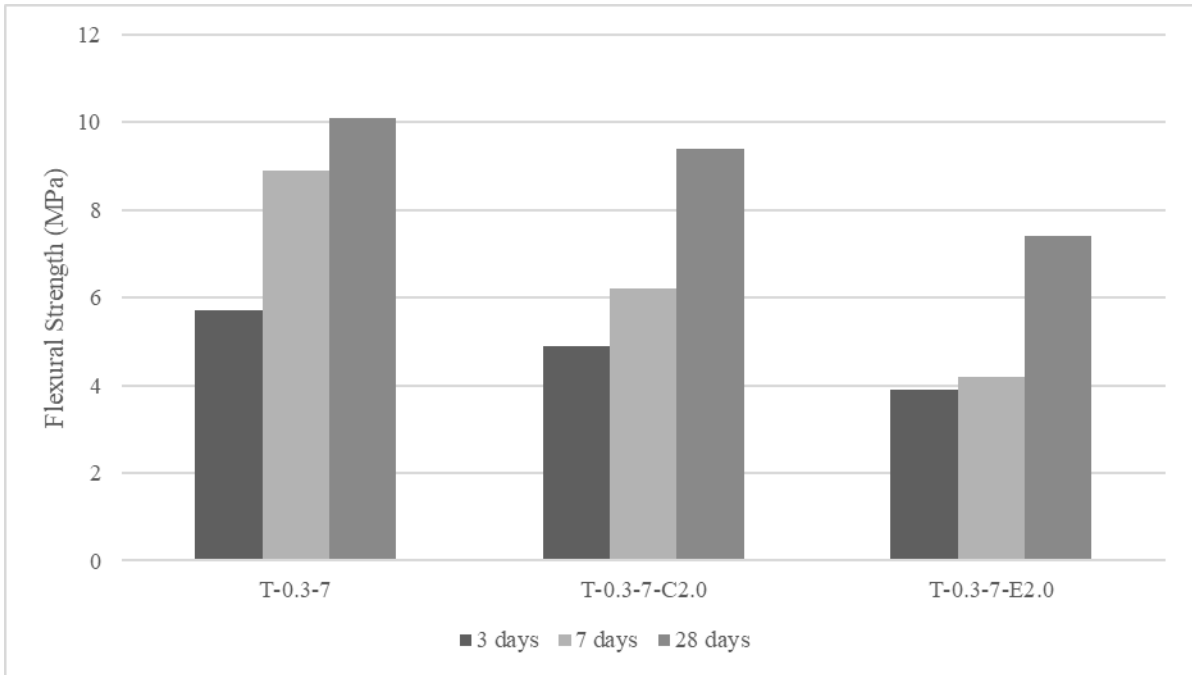


Figure 30 Flexural strength results for selecting the SP type in the ternary system.

The compressive strength results are shown in Figure 31. SP usage decreased the compressive strength of the samples. The sample T-0.3-7-C2.0 generally higher compressive strength than the sample T-0.3-7-E2.0. Since the composite containing SP (C) showed better performance, the polycarboxylate SP (C) was chosen as the SP type for the further steps.

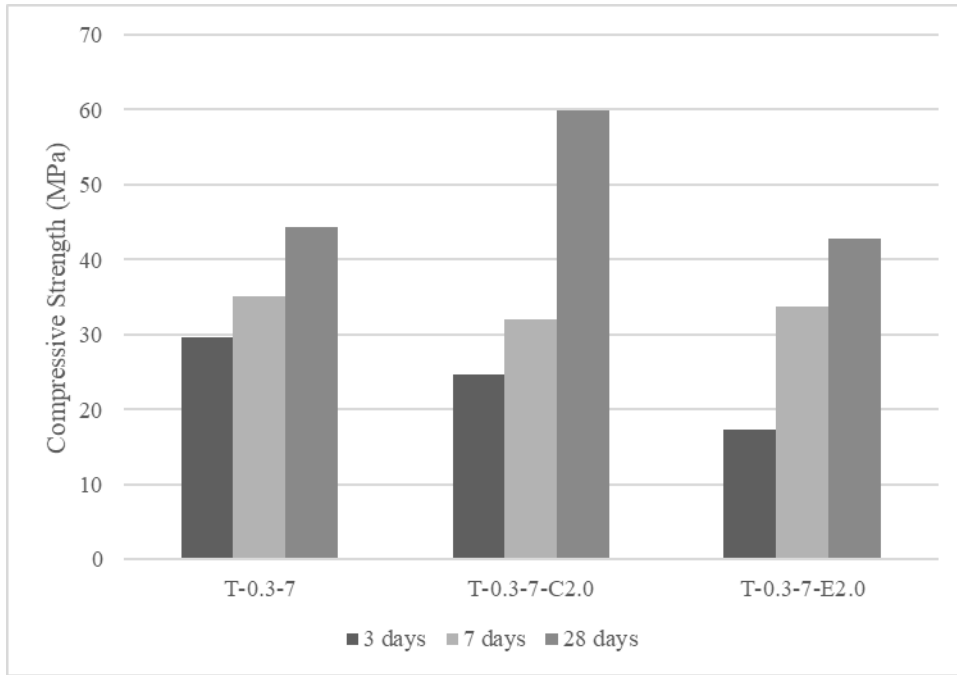


Figure 31 Compressive strength results for selecting the SP type in the ternary system.

4.2.2 Selection of Superplasticizer Content

5 different dosages of the SP were used to determine its optimum content in the ternary system, which were 0.5 %, 1.0 %, 1.5 %, 2.0 %, and 2.5 % of the total content of PC and CAC. As SP, the polycarboxylate-based one was used. The composites were compared in terms of mini slump diameter and strength results. The mix designs of the composites are given in Table 11.

Table 11 The mixture ratios for selecting the SP content.

Composite	CAC / (PC + CAC)	Cs / CAC (%)	C / (Binder + C)	S / Binder	SP(C) / (PC + CAC) (%)	W / Binder
T-0.3-7-C0.5	0.30	7.00	0.40	1.25	0.50	0.45
T-0.3-7-C1.0					1.00	
T-0.3-7-C1.5					1.50	
T-0.3-7-C2.0					2.00	
T-0.3-7-C2.5					2.50	

The mini slump flow diameters of the composites are given in Table 12. The ratios of 0.5 % and 1.0 % for the SP did not satisfy the self-compacting ability, and for the ratio of 1.5 %, the flow diameter of the mixture was at the minimum of the self-compacting limit stated in EFNARC [19] When the SP content was increased to 2 %, it satisfied self-compactability without segregation. For the higher content than 2 %, the mixtures showed high segregation. The sample T-0.3-7-C0.5 had voids on it due to the low flowability (Figure 32).

Table 12 The flow diameter of the mixtures.

Composite	Flow D. (cm)
T-0.3-7-C0.5	-
T-0.3-7-C1.0	17.5
T-0.3-7-C1.5	25.5
T-0.3-7-C2.0	33.8
T-0.3-7-C2.5	35.5



Figure 32 The composite T-0.3-7-C0.5.



Figure 33 Mini slump tests for the composites a) T-0.3-7-C0.5 b) T-0.3-7-C1.0 c) T-0.3-7-C2

The flexural strength results are shown in Figure 34. For 3 days, the sample T-0.3-7-C2.0 had the highest flexural strength with 4.9 MPa, and the flexural strength results at the 7th and 28th days were comparable up to 2 % of SP/(PC+CAC) ratio. For the composites having higher than 2 % of SP, it was determined that the flexural strength was lower than the others.

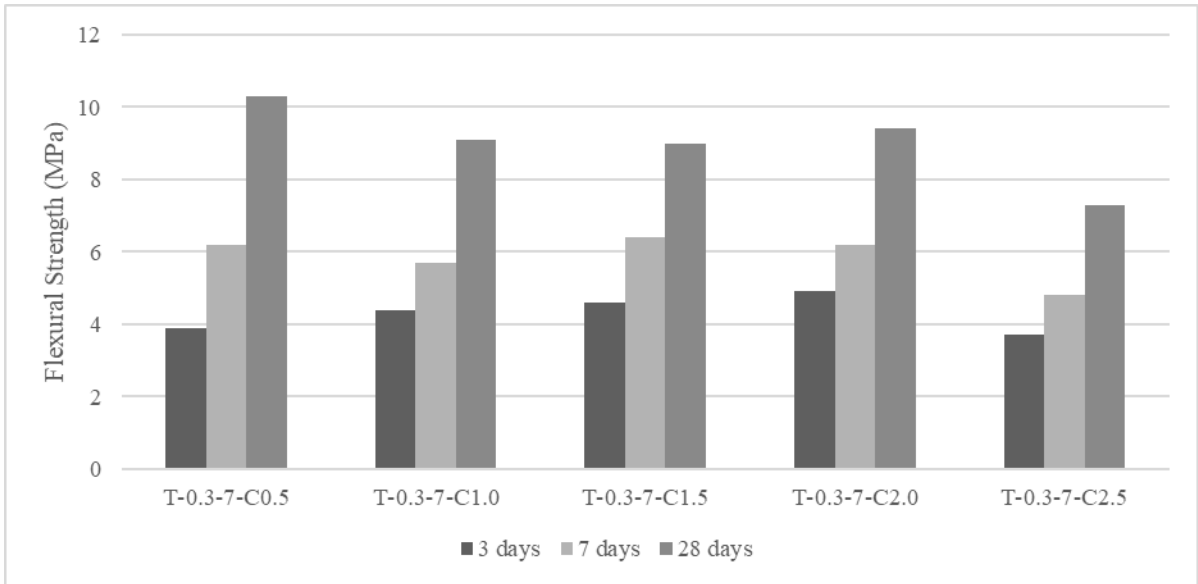


Figure 34 Flexural strength results for selecting the SP type in the ternary system,

The compressive strength results of the composites are given in Figure 35. A similar trend to the flexural strength was observed for the compressive strength results. The compressive strengths ranged between 20.9 - 24.6, 32.0 - 33.6, and 49.8 - 59.9 MPa for the 3rd, 7th, and 28th days, respectively except for T-0.3-7-C2.5. Its results were considerably lower than the others due to its higher segregation.

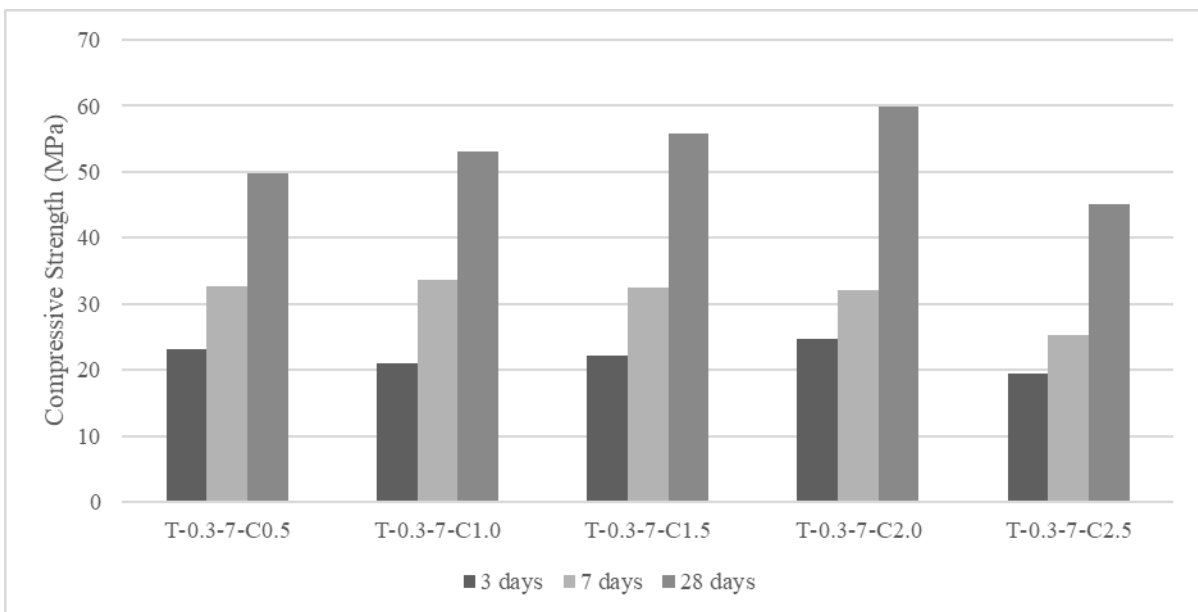


Figure 35 Compressive strength results for selecting the SP type in the ternary system.

The best results were obtained from the sample T-0.3-7-C2.0 for self-compacting ability. Even though the flexural and compressive strengths were comparable, the flow diameter from the mini slump test is the most important parameter for this section. T-0.3-7-C2.5 sample has the highest flow diameter. However, it showed very high segregation, and correspondingly, the mechanical test results were clearly lower than the sample T-0.3-7-C2.0. For these reasons, the SP/(PC+CAC) ratio was chosen as 2 %.

4.3. Selection of Set Retarders

Since a considerable content of CAC is present in the ternary system, the setting time can be very low. For a proper application of the flooring, it is very important not to have cold joints between the castings due to rapid setting. Therefore, it is crucial to incorporate suitable set retarders into the composites for suitable dosages. In this study, citric acid (CA) and potassium sodium tartrate (Rochelle salt – RS) were used as set retarders to increase initial setting time [25–27]. The Rochelle salt was one of the set retarders used to modify the setting time of calcium aluminate cements [28]. The other one was citric acid. In the study of Purnumo et al. [27], it was found that the optimum content of the citric acid with respect to cement amount was 0.45 % considering the strength, setting time, and workability points of view. Therefore, in this study, the minimum amount of the set retarders was decided at 0.5 % of the total content of PC and CAC. The RS and CA were utilized separately and binary to get a better solution for the rapid setting problem. The mix designs of the composites are given in Table 13.

Table 13 The mixture ratios for selecting the set retarder type and ratio.

Composite	PC/(PC+CAC)	CAC/(PC+CAC)	C _s /CAC (%)	C/(Binder + C)	S/Binder	SP/(PC+CAC) (%)	CA/(PC+CAC) (%)	RS/(PC+CAC) (%)	W/Binder
T-0.3-7-C2.0	0.70	0.30	7.00	0.40	1.25	2.00	0.00	0.00	0.45
T-0.3-7-C2.0-0:0.5							0.00	0.50	
T-0.3-7-C2.0-0:1.0							0.00	1.00	
T-0.3-7-C2.0-0.5:0							0.50	0.00	
T-0.3-7-C2.0-0.5:0.5							0.50	0.50	
T-0.3-7-C2.0-0.5:1.0							0.50	1.00	
T-0.3-7-C2.0-1.0:0							1.00	0.00	
T-0.3-7-C2.0-1.0:0.5							1.00	0.50	
T-0.3-7-C2.0-1.0:1.0							1.00	1.00	

The fresh properties of the composites are given in Figure 36. The self-compacting mortar obtained from the previous section (the composite T-0.3-7-C2.0) had 10 minutes for initial setting time and 33.8 cm for flow diameter obtained from the mini slump test. Using RS with 0.5 % and 1.0 % of the total content of PC and CAC, the initial setting time was increased by 150 % and 400 %, and for 0.5 % usage, decreased the flowability by 5.3 % but 1.0 % RS usage increased the flowability by 9.5 %. However, the same amount of single usage of CA clearly decreased the self-compacting ability. Belhadi et al. [29] discovered that CA caused a hydration delay, primarily by preventing the formation of the ettringite and amorphous phases as well as the dissolution of ye'elimitite and anhydrite. As a result, hydrates consumed less polycarboxylate ether, allowing the polycarboxylate ether dispersion efficiency to be maintained over time. For that reason, CA-containing samples had lower flow diameters. This could be observed in the composites having binary set retarder combinations, as well.

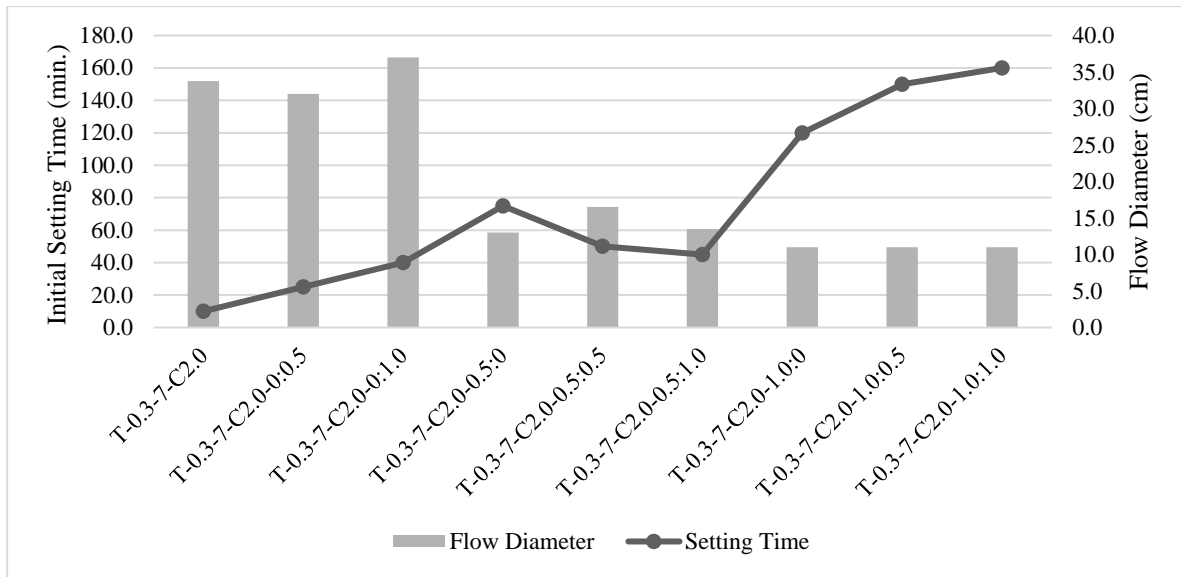


Figure 36 Fresh properties of the mixtures for selecting the set retarder type and content.

The flexural strengths of the samples are given in Figure 37. As seen, the larger quantities of set retarders decreased the flexural strength. Also, the samples containing RS only showed higher flexural strength compared to the ones with CA only and CA and RS used together. The base sample without retarder, T-0.3-7-C2.0, had 4.9, 6.2, and 9.4 MPa flexural strengths at the 3rd, 7th, and 28th days, but the flexural strength results dropped to 4.0, 4.2 and 6.4 respectively with the addition of 1 % RS. For the 0.5 % use of CA, the composites had higher flexural strength results than the one having 0.5 % RS. However, when the dosage was increased to 1 %, the composite T-0.3-7-C2.0-0:1.0 showed better performance than the composite T-0.3-7-C2.0-1.0:0. The flexural strength results of the composite T-0.3-7-C2.0-0.5:0.5 were between these of the composites T-0.3-7-C2.0-0:1.0 and T-0.3-7-C2.0-1.0:0.

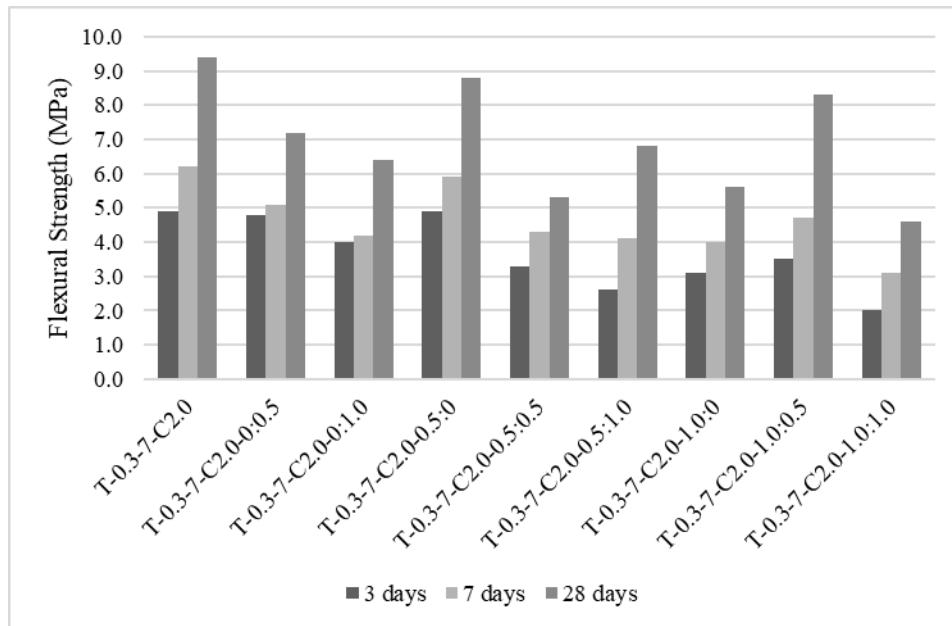


Figure 37 Flexural strength results for the selection of set retarder

The compressive strength of the mixtures decreased with the increase of set retarder content. The strength reduction was higher for CA-containing samples than that for the RS-containing samples. In another research [27], when CA content was above 0.45 %, the compressive strength of the sample decreased as well. Also, Kastiukas et al. [26] researched the effect of the CA on PC/CAC blended systems by 0.5 %, 1.0%, and 3.0 %, by weight and reported that CA usage at those ratios decreased the strength of the mortars since the dissolution of C_3S and C_3A slows down considerably.

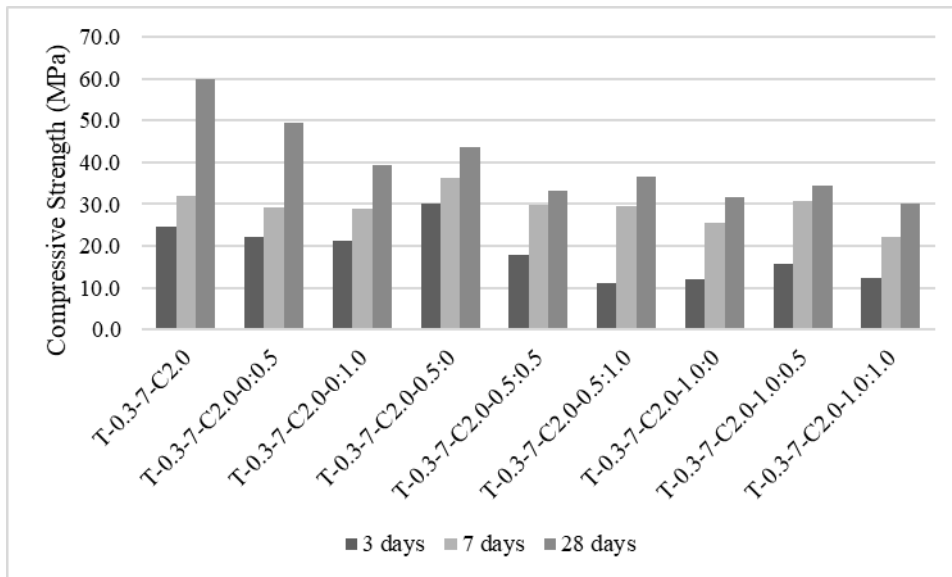


Figure 38 Flexural strength results for the selection of set retarder.

Since the CA-containing mixtures did not show self-compacting ability, those mixtures were eliminated. Also, the composite T-0.3-7-C2.0-0:0.5 had 25 minutes of initial setting time while the composite T-3.0-7-C2.0-0:1.0 had 40 minutes of initial setting time, the set retarder for the self-compacting ternary system chosen as 1 % RS of the total of PC and CAC, by weight.

4.4. Selection of the Shrinkage Reducing Materials

The volume stability of ternary systems used as flooring can be critical since it has a very high surface with a very low thickness. Its surface can lead to excess evaporation of the water utilized in the system, causing shrinkage. In addition, with its low thickness and typical low tensile strength, the flooring can have trouble with shrinkage cracks [1]. To reduce the shrinkage of the system, commercially obtained shrinkage-reducing admixture (SRA) and polypropylene fibers (PPF) were used in this section [5,9]. The SRA content was determined from the technical data sheet of the admixture and used with 0.5 % and 1 %, by weight of total content of the PC and CAC while PPF was used as 1 % and 2 % by weight of total weight of the mixture according to the studies [7]. It was stated that the typical usage rate of PPF in self-compacting concrete was between 0.3 % and 1.2 % by weight of the concrete [7,30]. Besides the ternary systems, for comparison purposes, two Portland cement-based systems were also tested in terms of shrinkage, strength, and fresh-state state properties. For

the samples REF and PC-C2.0, CEM-I and CEM-II A/LL cements were used, respectively. The mixture designs are given in Table 14.

Table 14 Mixture proportions for selecting the shrinkage reducing materials.

Composite	PC / (PC + CAC)	CAC / (PC + CAC)	Cs / CAC (%)	C / (Binder + C)	S / Binder	SP / (PC + CAC) (%)	RS / (PC + CAC) (%)	SRA / (PC + CAC) (%)	PPF / Total weight (%)	W / Binder
T-0.3-7-C2.0-0:1.0	0.70	0.30	7.00	0.40	1.25	2.00	1.00	0.00	0.00	0.45
T-0.3-7-C2.0-0:1.0-0.5:0								0.50	0.00	
T-0.3-7-C2.0-0:1.0-1.0:0								1.00	0.00	
T-0.3-7-C2.0-0:1.0-0:1.0								0.00	1.00	
T-0.3-7-C2.0-0:1.0-0:2.0								0.00	2.00	
T-0.3-7-C2.0-0:1.0-0.5:1.0								0.50	1.00	
T-0.3-7-C2.0-0:1.0-1.0:2.0								1.00	2.00	
REF	1.00	0.00	0.00				0.00	0.00	0.00	
PC-C2.0										

The fresh properties of the samples are given in Figure 39. The composite T-0.3-7-C2.0-0:1.0 had a 37.0 cm-flow diameter while those for the composites T-0.3-7-C2.0-0:1.0-0.5:1.0 and T-0.3-7-C2.0-0:1.0-0:1.0 had 34.0 cm and 35.8 cm, respectively. On one hand, using SRA at 0.5 % and 1.0 % ratios reduced the initial setting time from 40 minutes to 10 minutes. On the other hand, PPF usage of 1 % and 2 % reduced setting time to 15 minutes and 20 minutes for the composites, respectively. The combined usage of SRA and PPF in the mixtures T-0.3-7-C2.0-0:1.0-0.5:1.0 and T-0.3-7-C2.0-0:1.0-1.0:2.0 decreased the initial setting time to 20 and 25 minutes, respectively. As expected, the Portland cement-based systems had very longer setting times due to the amount of superplasticizer incorporated into them. However, in order to show the setting time results on Figure 4.14, they were assumed to be 120 minutes.

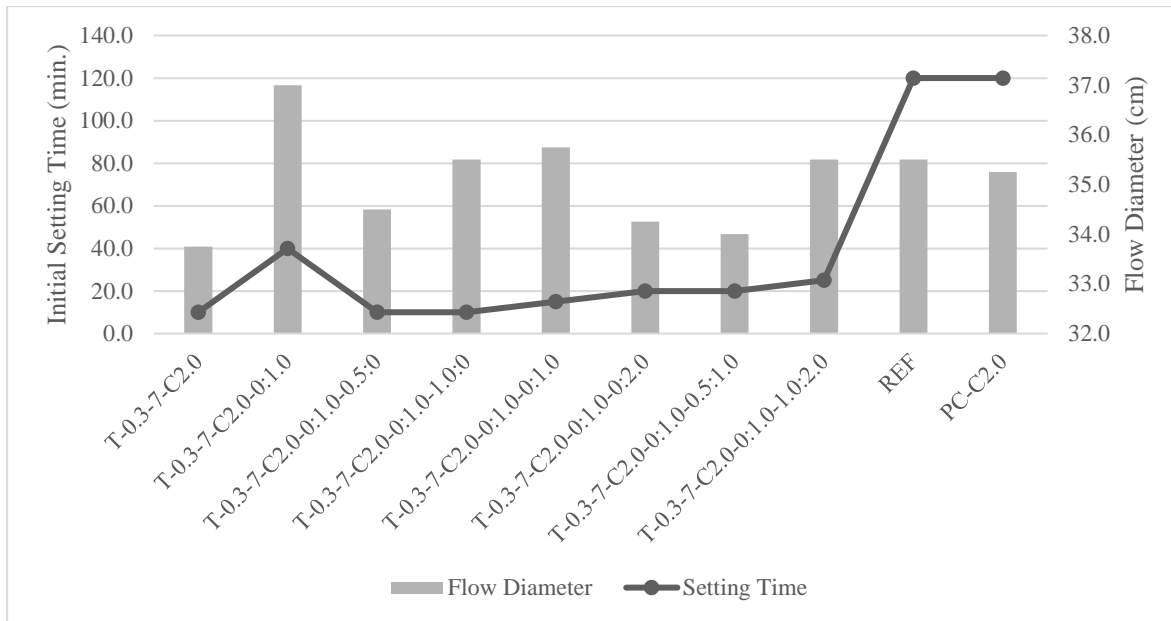


Figure 39 Fresh properties of the samples for selection of the shrinkage reducing materials.

The flexural strengths of the mixtures are given in Figure 40. The SRA use in the mixtures increased the flexural strength, due probably to lower setting time. The flexural strengths and initial setting times of the composites T-0.3-7-C2.0-0:1.0-0.5:0 and T-0.3-7-C2.0-0:1.0-1.0:0 were similar to those of the composite T-0.3-7-C2.0. The 2 % usage of PPF increased the flexural strength of the mixture providing the bridging effect [31]. However, the composites having 1 % PPF do not show sufficient performance, and their strength results were lower. The air entrapped due to the addition of fibers [32] can be the reason for the strength reduction. But, at higher dosages, the bridging effect could dominate and increase the strength. The combined usage of SRA and PPF results were lower than the only SRA-containing samples.

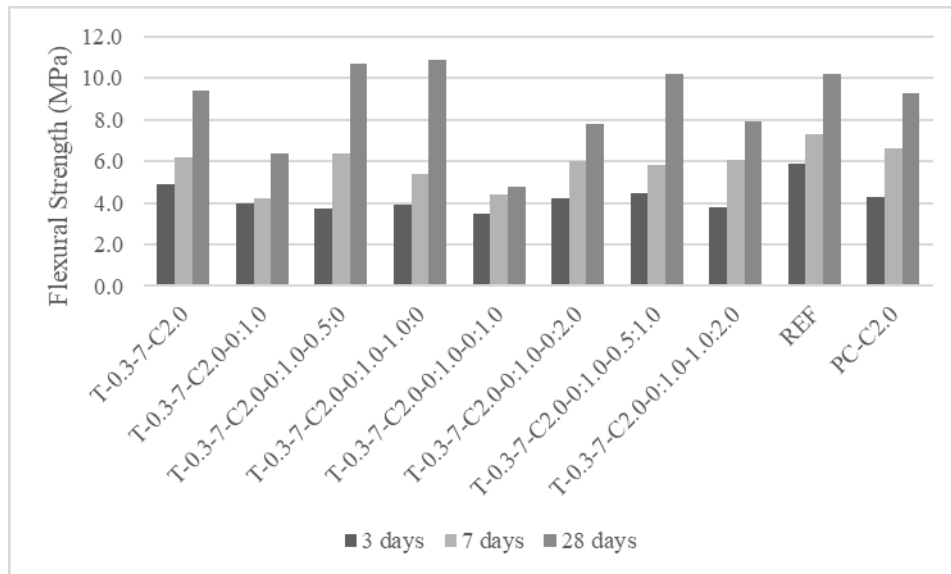


Figure 40 Flexural strength of the samples for selection of the shrinkage reducing materials.

The compressive strengths of the composites are given in Figure 41. Using SRA and PPF increased the compressive strength. Only the sample T-0.3-7-C2.0-0:1.0-0:1.0 yielded lower compressive strength results than T-0.3-7-C2.0-0:1.0. The maximum compressive strength results were obtained from REF and PC-C2.0 mixtures with 55.0 and 59.3 MPa at 28th days which did not include any amount of set retarders. The best compressive results were obtained from T-0.3-7-C2.0-0:1.0-0.5:1.0 with 23.7, 33.9, and 49.3 MPa at 3rd, 7th and 28th days respectively for ternary system-based composites. Using PPF increased the flexural strength but its effect on the compressive strength is tolerable which is similar to the study done by Yousefieh et al. [33] and Rani and Priyanka [7].

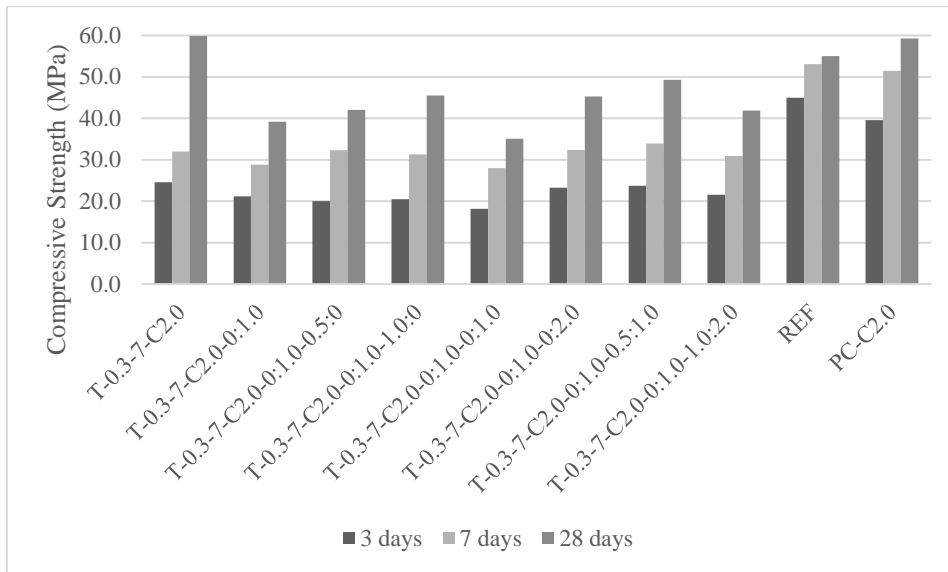


Figure 41 Compressive strength of the samples for selection of the shrinkage reducing materials.

The length changes of the composites are given in Figure 42. The samples REF and PC-C2.0 showed similar shrinkage results generally, their length change values were in the range of $25 \mu\epsilon$ after 2 weeks which exceeds the ultimate shrinkage limit stated in ASTM-C-596. For the REF sample, the shrinkage result at 28 days was $641 \mu\epsilon$. The sample T-0.3-7-C2.0-0:1.0 had lower shrinkage than the sample REF, on the 7th day, the difference between the results was negligible. However, after 14 days, the length change of the composite T-0.3-7-C2.0-0:1.0 was 35.7 % lower. Not only the composite T-0.3-7-C2.0-0:1.0 showed lower shrinkage but also its shrinkage rate was generally lower than that of the composite REF and the limit stated ASTM [23]. The SRA-containing samples T-0.3-7-C2.0-0:1.0-0.5:0 and T-0.3-7-C2.0-0:1.0-1.0:0 showed similar shrinkage rates and the length change results were 198 and 199 $\mu\epsilon$ at 7th day and 253 and 304 $\mu\epsilon$ at 28th day, respectively which were considerably lower than those of the composite T-0.3-7-C2.0-0:1.0 which are 250 and 410 $\mu\epsilon$. The PPF usage reduced the shrinkage at early ages but at later ages, they did not provide the sufficient volume stability. The shrinkage results of the composites having both SRA and PPFs were higher than those of the composites containing SRA only. For shrinkage, the length of fibers might not be so low that they might not affect the volume stability [5]

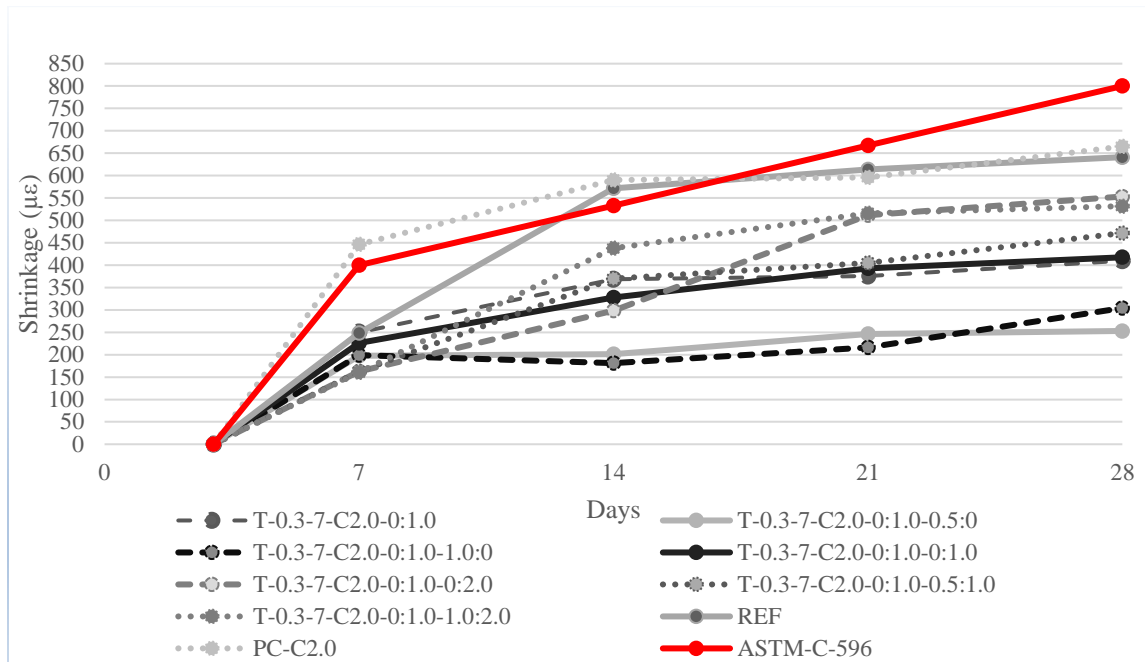


Figure 42 Drying shrinkage results of the samples for selection of the shrinkage reducing materials.

The best results were obtained from the composites T-0.3-7-C2.0-0:1.0-0.5:0 and T-0.3-7-C2.0-0:1.0-1.0:0 but the initial setting time was decreased by 75 % with the use of SRA. Therefore, these 2 mixtures were selected to optimize to have a longer setting time and, correspondingly, application time with low shrinkage capacity.

4.5. Optimizing the Final Design

The composites T-0.3-7-C2.0-0:1.0-0.5:0 and T-0.3-7-C2.0-0:1.0-1.0:0 having low setting time made their application limited and harder to use on larger areas. To increase the setting time, the retarder (RS) amount increased to 1.5 % for both, and 2.0 % ratio was also tried for T-0.3-7-C2.0-0:1.0-1.0:0. The mixture designs are given in Table 15.

Table 15 The mixture design for the optimization process.

Composites	PC / (PC + CAC)	CAC / (PC + CAC)	Cs / CAC (%)	C / (Binder + C)	S / Binder	SP / (PC + CAC) (%)	RS / (PC + CAC) (%)	SRA / (PC+CAC) (%)	W / Binder
T-0.3-7-C2.0-0:1.0-0.5:0	0.70	0.30	7.00	0.40	1.25	2.00	1.00	0.50	0.45
T-0.3-7-C2.0-0:1.5-0.5:0							1.50	0.50	
T-0.3-7-C2.0-0:1.0-1.0:0							1.00	1.00	
T-0.3-7-C2.0-0:1.5-1.0:0							1.50	1.00	
T-0.3-7-C2.0-0:2.0-1.0:0							2.00	1.00	

Increasing the amount of RS increased the flow diameter and setting time for all the mixtures. However, 2.0 % usage of the RS caused segregation on the composites T-0.3-7-C2.0-0:2.0-:1.0:0 and the setting time increased to 120 minutes. The samples T-0.3-7-C2.0-0:1.5-:1.0:0 and T-0.3-7-C2.0-0:1.5-:0.5:0 had setting times of 40 and 30 minutes, respectively.

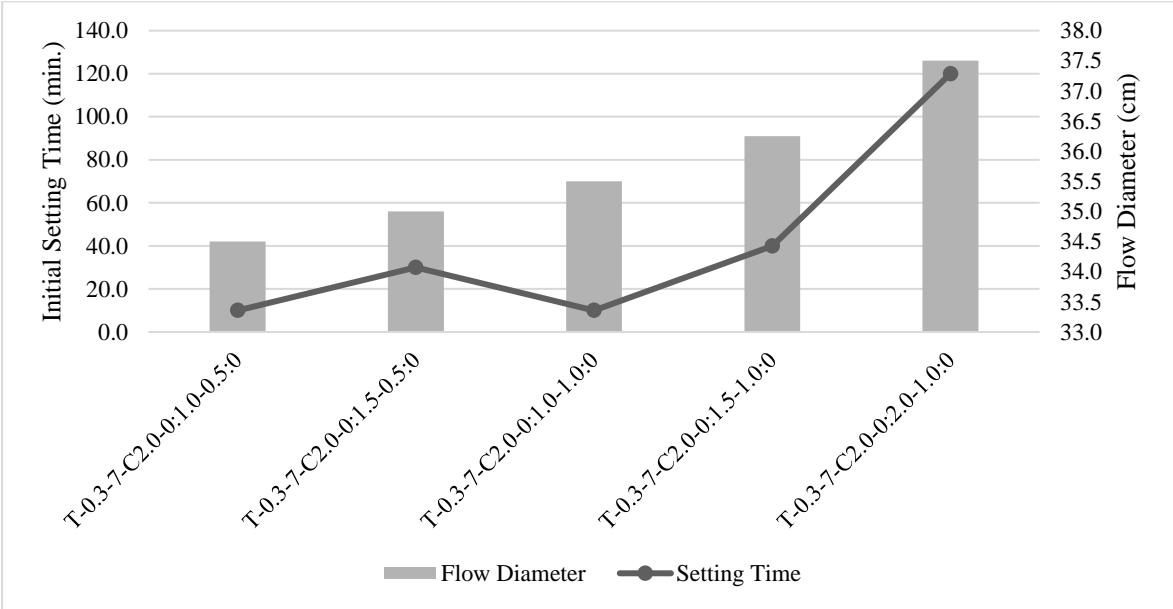


Figure 43 Fresh properties of the optimization section

The flexural and compressive strengths are given in Figure 44 and Figure 45, respectively. Higher amounts of RS decreased the flexural and compressive strengths of the samples. The composite T-0.3-7-C2.0-0:1.5-1.0:0 mixture had higher strength compared to the composite T-0.3-7-C2.0-0:1.5-:0.5:0 and the lowest results were obtained from the composite T-0.3-7-C2.0-0:2.0-:1.0:0.

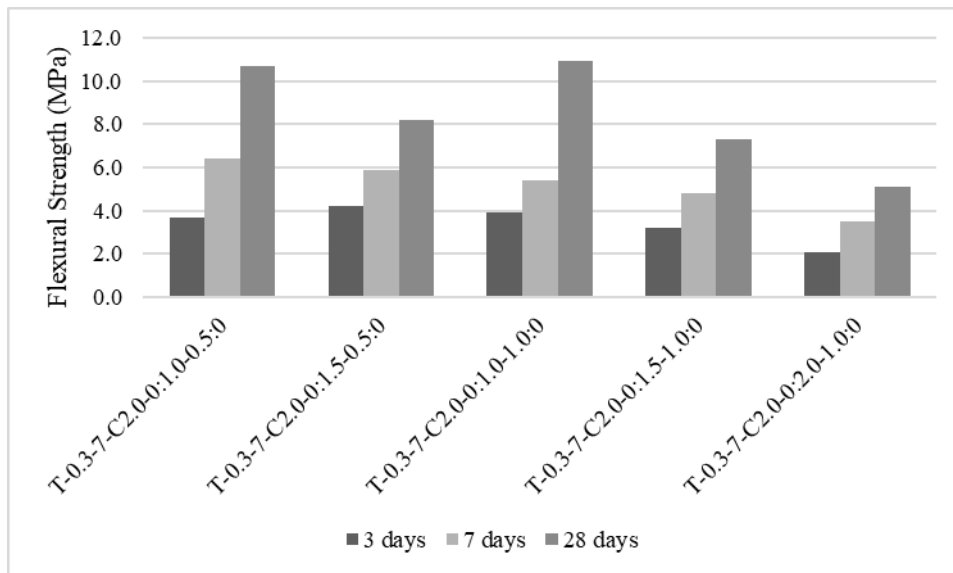


Figure 44 Flexural strengths of the optimization of the mixture.

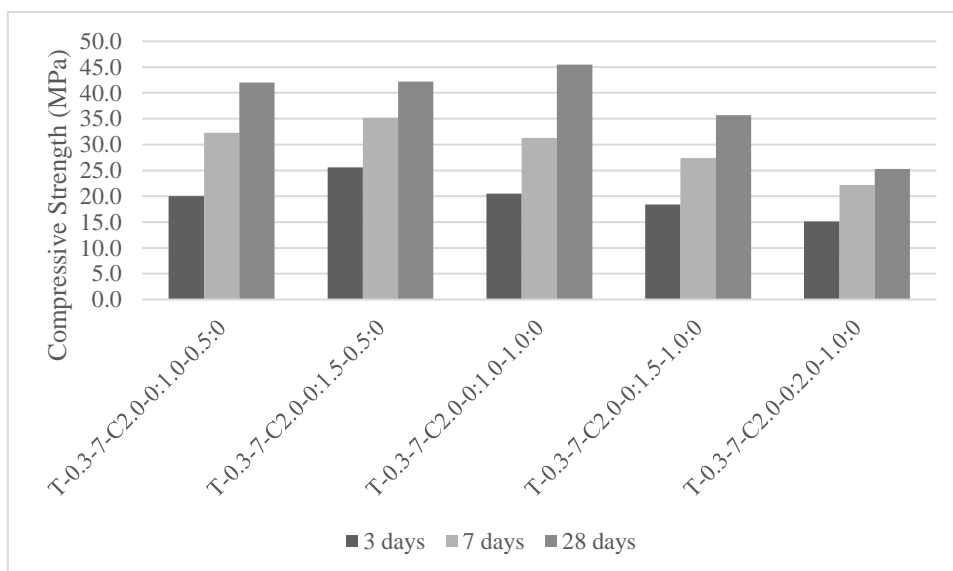


Figure 45 Compressive strengths of the optimization of the mixture.

For the same amount of SRA, the higher content of RS caused higher shrinkage rates. For 1.0 % of RS content, the composite with lower SRA content had a lower shrinkage result but when the RS content increased to 1.5 %, higher SRA content resulted in lower shrinkage.

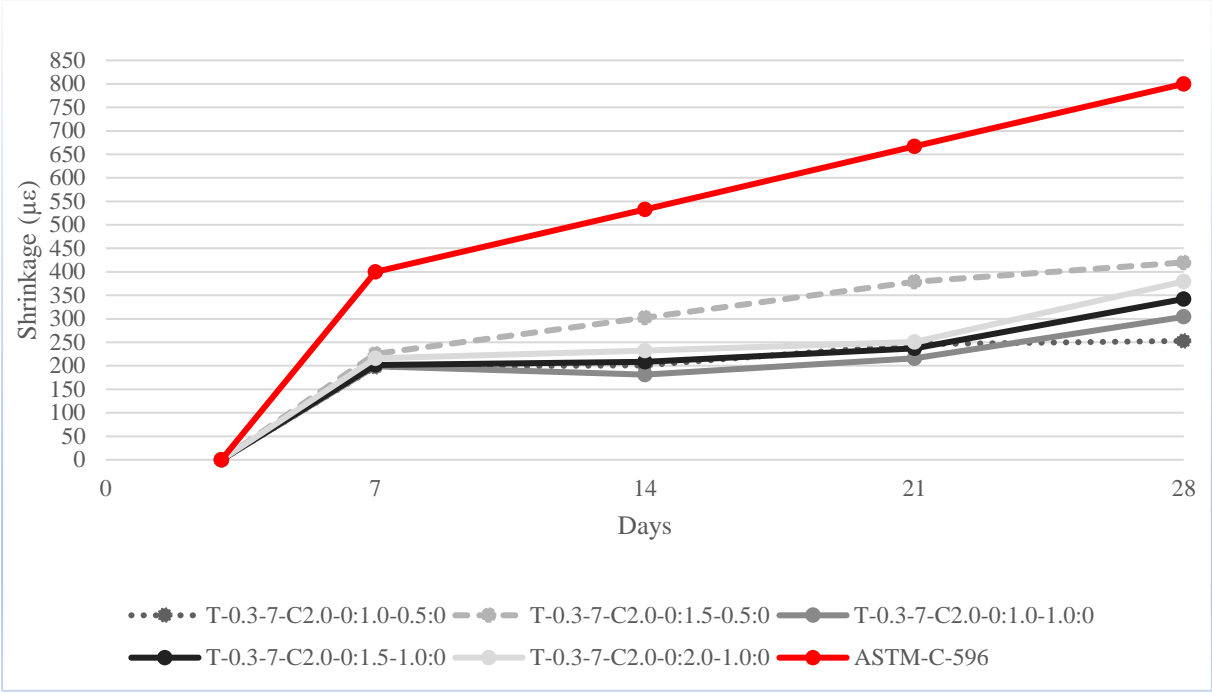


Figure 46 Drying shrinkage results of the optimizing the final design.

Due to the setting time exceeding 30 minutes and high strength values, the composite T-0.3-7-C2.0-0:1.5-1.0:0 was chosen as the final design. But for the applications which should be done in shorter times, the composites T-0.3-7-C2.0-0:1.0-0.5:0 and T-0.3-7-C2.0-0:1.0-1.0:0 can be used as well.

5. CONCLUSION AND RECOMMENDATIONS

5.1 Conclusion

Within the scope of the study, it was aimed that obtain a self-compacting ternary system with low shrinkage capacity to satisfy at least 25 cm flow diameter, 2.5 MPa flexural strength and 15.0 MPa compressive strength on 3rd day, 6.0 MPa flexural strength and 30.0 MPa compressive strength on 28th day with lower shrinkage capacity than the ultimate shrinkage value from ASTM-C 596 and the self-compacting reference sample containing Portland cement as the main binder and the results of the study achieved that goal by having 46.5 % and 68.3 % lower shrinkage results than using self-compacting mortar with Portland cement system and ultimate shrinkage limit in ASTM-C 596. The following conclusions have been observed from the test results of this study:

- The CAC/(PC+CAC) ratios from 0.1 to 0.4 and Cs/CAC ratios from 0.07 to 0.09 increased the flexural and compressive strength of the mortar in ternary systems. The flexural and compressive strength of the ternary system increases up to 0.3 CAC/(PC+CAC) ratio, then the strength of the composites decreases for Cs/CAC content 0.07.
- The flexural and compressive strength of the system increases up to 0.07 Cs/CAC ratio, afterward the strength reduction is observed.
- The compressive strength/flexural strength ratio of ternary systems is higher than the cementitious system.
- The polycarboxylate-based SP showed better performance than the naphthalene-based SP in the PC-CAC-Cs ternary system.
- Up to 2 % of SP/(PC+CAC) ratio, polycarboxylate-based SP increases the flowability with acceptable strength reduction but after 2 % segregation is observed with higher strength reductions.
- When citric acid and polycarboxylate SP are used together, SP loses its water-reducing characteristics due to their possible interaction.
- Higher ratios of RS/(PC+CAC) than 1.5 % caused segregation and lowered the flexural and compressive strength considerably.
- The SRA reduced the setting time of the self-compacting ternary system containing the polycarboxylate-based SP.

- The use of the SRA reduced the shrinkage, but that of PPF had no noticeable effect on shrinkage.
- 1 and 2 % of PPF usage, by total weight increased the flexural strength, but its effect on compressive strength was negligible.
- The use of PPF did not improve the volume stability of the composites.

5.2 Recommendations

The following suggestions for further research may be helpful:

- The compressive strength/flexural strength ratio of ternary systems is higher than the cementitious systems. The precise cause of this is unknown from the literature research; however, the microstructure of the hydration products of ternary systems, particularly ettringite, may hold the key. The reason for that should be investigated in advanced studies.
- In order to obtain a composite showing higher performance than the final composite determined in this study, different types of chemical admixtures, fibers and calcium sulfate sources can be tried in the production with various water/binder ratios.
- In order to be sure that the composite finalized will work in long term properly, the durability tests, such as freezing and thawing, sulfate attack, alkali-silica reaction tests should be conducted on these composites.
- To determine the mix design of the final composite is suitable for large scale productions, the amount of the mixture should be increased as much as possible and it should be tried in real-life applications.

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