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Effect of superplasticizer on the fresh and hardened properties of mortars prepared with different types of cement produced from the same clinker

Süperakışkanlaştırıcının aynı klinkerden üretilen farklı çimento türleri ile hazırlanan harçların taze ve sertleşmiş özelliklerine etkisi

Derya ÖVER[1], Nesil ÖZBAKAN[1], Sinan AÇIKYOL[2], Abdulkadir BAKIRCI[3]*

[1]Department of Civil Engineering, Faculty of Engineering, Eskisehir Technical University, Eskisehir, Turkey
deryaover@eskisehir.edu.tr, nesil_ozbakan@eskisehir.edu.tr
[2]Istanbul Metropolitan Municipality, Istanbul, Turkey.
acikyolsinan@gmail.com
[3]Directorate of Environment, Urbanisation And Climate Change, Sanlıurfa, Turkey.
abdulkadirbakirci@outlook.com

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Abstract

In recent years, extensive research has been done on using superplasticizers as a chemical additive in concrete. However, these studies generally compare the effects of superplasticizers on cements of different compositions. This paper presents data from laboratory tests of mortars prepared with various cement types produced from the same clinker at the same plant. Increasing dosages of superplasticizers (i.e. 0.8%, 1.0% and 1.2%) were added to CEMI, CEMII, CEMIII and CEMIV cements produced from the same clinker. The effects of superplasticizers on the workability and strength were analyzed by fresh and hardened mortar tests. The results showed that clinker played the main role when superplasticizer was used. Other modifications made to that cement, had a minor effect only. The use of superplasticizers improved cement behavior, increased flowability and reduced water requirement, regardless of cement type. Moreover, the flexural and compressive strengths increased since the addition of superplasticizers improved the compressibility of the mortar.

Keywords: Cement, Clinker, Superplasticizers, Fresh properties, Hardened properties

Öz

Son yıllarda, süper akışkanlaştırıcıların betonda kimyasal katkı olarak kullanılması konusunda birçok araştırma yapılmıştır. Ancak bu çalışmalar genellikle süper akışkanlaştırıcıların farklı bileşimlerdeki çimentolar üzerindeki etkilerini karşılaştırmaktadır. Bu makale, aynı fabrikada aynı klinkerden üretilen farklı çimento türleri ile hazırlanan harçların laboratuvar testlerinden elde edilen verileri sunmaktadır. Aynı klinkerden üretilen CEMI, CEMII, CEMIII ve CEMIV çimentolarına artan dozlarda süper akışkanlaştırıcı (%0.8, %1.0 ve %1.2) eklenmiştir. Süper akışkanlaştırıcı ilavesinin işlenebilirlik ve dayanım üzerindeki etkileri taze ve sertleştirilmiş harç testleri ile analiz edilmiştir. Sonuçlar, süper akışkanlaştırıcı kullanıldığında klinkerin ana rolü oynadığını göstermektedir. Çimentoda yapılan diğer modifikasyonların yalnızca küçük bir etkisi olmuştur. Süper akışkanlaştırıcı kullanımı, çimento tipinden bağımsız olarak çimento davranışını iyileştirmiş, akışkanlığı artırmış ve su gereksinimini azaltmıştır. Ayrıca, süper akışkanlaştırıcı ilavesi harcın sıkıştırılabilirliğini geliştirdiği için eğilme ve basınç dayanımları artmıştır.

Anahtar kelimeler: Çimento, Klinker, Süperakışkanlaştırıcı, Taze özellikler, Sertleşmiş özellikler

1 Introduction

Chemical admixtures have become a vital element of conventional concrete, which might be one of the primary factors contributing to concrete's continued status as the most widely utilized building material in the construction industry. Generally, every important property of fresh concrete, such as consistency, workability, and cohesion, as well as strength and durability of hardened concrete depends on the amount of water, type of cement, and clinker phases of the cement.

The water demand, which is the amount of water required to prepare a standard consistency cement paste [1], has a great impact on the overall quality of the concrete. The water demands of Portland cement types may differ from each other. The chemical, physical and mineralogical properties of cement play an important role in that demand. Grinding of the clinker to different finenesses or using different proportions of mineral additives in the cement can lead to a change in the water requirements of the cement [2].

The quantities of the four primary clinker phases, along with the minor clinker phases, exert a notable influence on various processes, including setting time, heat evolution rate, volume change, and strength development [3]. Portland cement clinker, which is produced under a relatively wide range of limits, can show different properties such as compressive strength, setting time, and hydration temperature depending on its fineness [4]. Studies using cements with the same clinker composition can help explain the differences in cement properties. In literature, various cement properties were evaluated using cements produced from the same clinker. Mardani-Aghabaglou et al. [5] conducted a study utilizing two distinct cement types produced from the same clinker to investigate the influence of gypsum type. The research revealed that irrespective of the cement type employed, the final strengths of the mixes were remarkably similar. Vikan et al. [6] investigated the effect of clinker composition and cement fineness on flow resistance. Their study revealed that there is a correlation between flow resistance and cement fineness for cements produced from the same clinker. Hosten and Fidan [7] evaluated the properties of

*Corresponding author/Yazışılan Yazar

cements made from the same clinker and having approximately the same fineness to understand the influence of cement grinding systems on cement properties. The study's findings showed that while the grinding systems had no noticeable effect on water demand, they had a significant effect on setting times and strength development. On the other hand, Turkel et al. [8] compared acid resistance of different type of cements made from same clinker. In a similar manner, the corrosion behavior of steel and cement made from same clinker in concrete was investigated [9]. In addition, some studies have been carried out to compare the sulfate resistance of different types of cement produced from the same clinker. For these cements, increasing the fineness of the cement increased the expansions [10] and the sulfate resistance of ordinary Portland cement (OPC). Moreover, it was noted that OPC was more sensitive to the w/c ratio compared to blended cements [11].

On the other hand, the addition of supplementary cementitious materials affects the water demand of blended compositions [12]. Various researches have been carried out to determine how partial replacement of clinker with supplementary cementitious materials can affect water demand. Compared to OPC, partial replacement of cement with fly ash reduces the amount of water needed to achieve a normal consistency [13–15]. Similarly, the cements containing slag require less water than the reference slag-free cement [16,17]. However, water requirement for normal consistency increases with increasing silica fume content [18,19]. Moreover, the use of trass can also increase the water demand [20].

There are numerous chemical admixtures accessible to enhance the workability of cement while simultaneously reducing water requirements. According to the definition of the American Concrete Institute, superplasticizers are used to considerably increase the slump without adding extra water or to drastically reduce water content without causing slump loss. [21]. They are widely used as a component of cement-based materials and significantly affect their properties. Superplasticizers, also called high range water-reducing admixtures, can reduce the mixing water requirement in a given concrete mix by three to four times compared to normal-range water-reducing admixtures [22]. The adsorption of superplasticizers onto cement particles is influenced by both the composition of the cement clinker and the characteristics of the superplasticizers themselves [23]. Adsorption of superplasticizers on the surface of cement diminishes the magnitude of the van der Waals forces holding the cement particles together by preventing the cement particles from getting too close to each other. The improved flowability of the mixture is a direct result of reducing the magnitude of these forces [24]. The fresh state properties and rheology of mortars produced with Portland cement can be altered or controlled by the addition of a superplasticizer. [25]. However, in some cases, incompatibility occurs between the cement and the admixture, and these incompatibilities may adversely affect the fresh and hardened characteristics of the mixture. The reaction between the superplasticizer and the C_3A phase of the clinker can cause compatibility problems [26]. Moreover, cement and superplasticizer compatibility is affected by the physical and chemical properties of the mineral additives added to cement [25]. It is a common approach to compare the effects of superplasticizers on cements of different compositions. Despite the important information that these studies can provide, they are prone to misinterpretation due to the many variables that may occur between cements [27]. It has been stated that even in cements made from same clinker, the grinding aid may affect the adsorption of superplasticizers and lead to different workabilities between the cements [28].

The clinker phases of the cement has a notable impact on numerous fresh and hardened properties of the mortar. The incorporation of chemical admixtures plays a crucial role in attaining the desired property goals of the mortar. Another factor of great importance to the overall quality of the mortar is the water demand. Within the scope of the study, the workability and strength properties of different types of cements produced from the same clinker and having the same w/c ratio are examined. Using the same w/c ratio while preparing different cement types will reduce the number of variables and contribute to a more meaningful comparison. In a similar manner, the use of cements produced from the same clinker can help explain the differences in cement properties. Another issue discussed in the study is the determination of the effect of superplasticizers on different types of cement produced from the same clinker. The clinker phase in cement has a significant impact on mortar properties. In contrast to most studies that compare the effects of superplasticizers on cements with different compositions, this study examines the impact of superplasticizers on mortars prepared with cements produced from the same clinker in order to reduce the number of variables that could arise between different cements. Reducing the differences that may be encountered between cements will contribute to the evaluation of the superplasticizer effect.

2 Experimental Methods

2.1 Materials

The study was carried out with four types of cement manufactured from the same clinker. Cements were produced from the same clinker and were supplied from a single company. An ordinary Portland cement and three different blended cement containing clinker, natural pozzolan and granulated blast furnace slag in different proportions were produced in accordance with TS EN 197-1 [29]. The compositions of the assessed cement samples according to standards are presented in Table 1. CEMII and CEMIII contained the lowest amount of natural pozzolan and granulated blast furnace slag, respectively, while CEMIV contained a higher amount of natural pozzolan compared to CEMII. The strength class of CEMI, CEMII, and CEMIII was 42.5R, and for CEMIV it was 32.5R. The physical and chemical properties of the cements are listed in Table 2 and Table 3, respectively. In accordance with the TS EN 197-1 standard [29], the initial setting time is over 60 minutes for CEMI, CEMII, and CEMIII cement with a strength class of 42.5R and over 75 minutes for CEMIV cement with a strength class of 32.5R. Moreover, CEMI and CEMIII cement meet limit values for loss on ignition and insoluble residue, and all four cement meet the requirements for sulfate and chloride content according to TS EN 197-1 [29]. X-ray diffraction (XRD) results of cements are given in Figure 1.

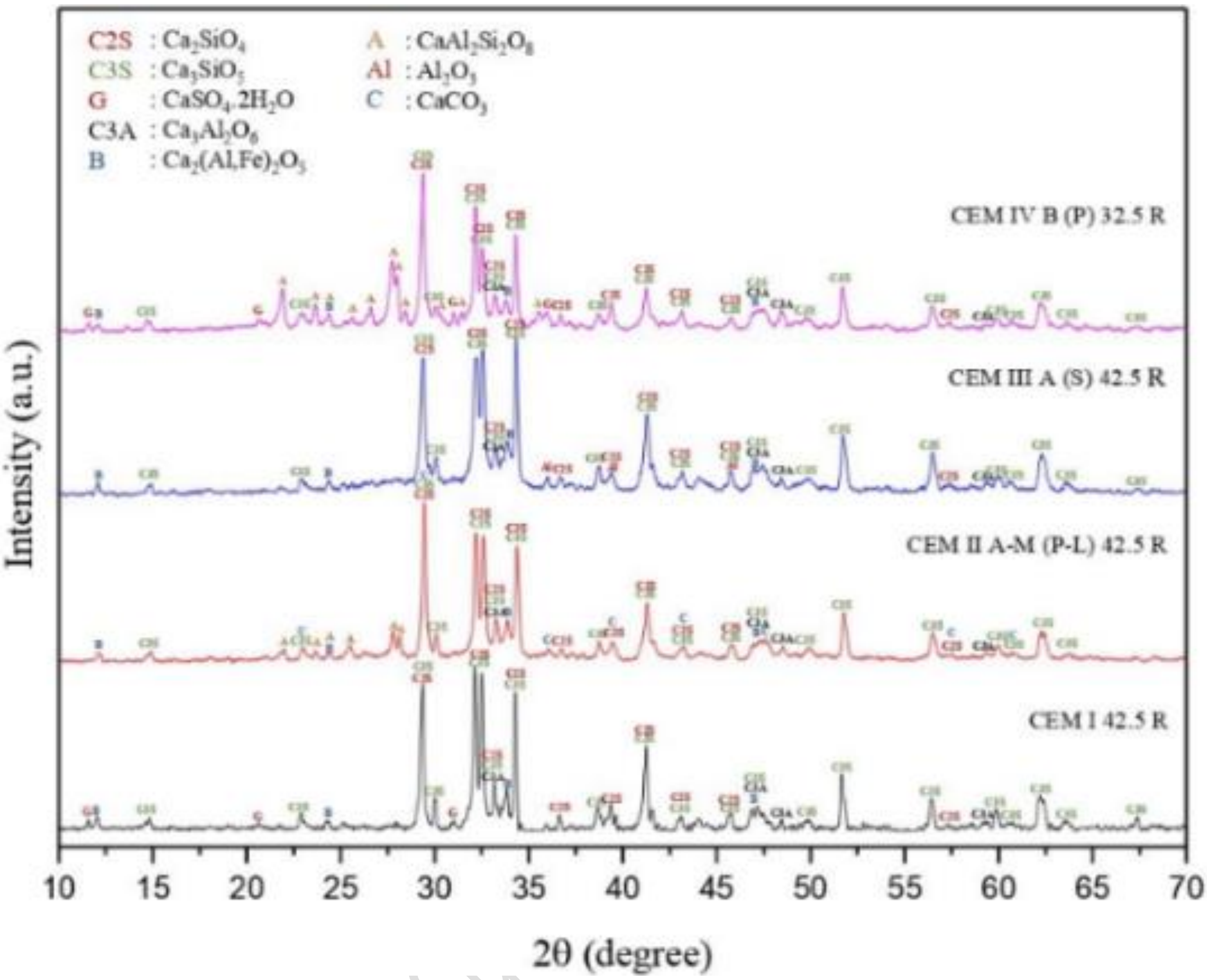


Figure 1. XRD results of the cements.

When the XRD patterns of cements produced from the same clinker were compared, it was observed that the phases were quite similar. While C3S (Ca3SiO5) and C2S (Ca2SiO4) were observed as the main phases in all cements, anorthite (CaAl2Si2O8) phases were observed as minor phases in CEMII and CEMIV cements containing pozzolan, and alumina (Al2O3) phases were observed in CEMIII cement containing slag.

Table 1. The compositions of assessed cement types.

Cement Type	Type	Composition (%)		
		Clinker	Natural Pozzolana	Blast Furnace Slag
CEMI 42.5R	Ordinary Portland Cement	95-100	-	-
CEMII/A-M (P-L) 42.5R	Portland Pozzolana Cement	80-94	6-20	-
CEMIII/A 42.5R	Blast Furnace Cement	35-64	-	36-65
CEMIV/B (P) 32.5R	Pozzolana Cement	45-64	36-55	-

Table 2. The physical properties of the cements.

Physical Properties	Specific Gravity, g/cm3	Specific Surface, cm2/g	Initial Setting Time, min	Soundness (Le Chatelier), mm
CEMI	3.16	3942	139	1
CEMII	3.1	4614	175	1
CEMIII	3.08	5610	143	1
CEMIV	2.92	5412	180	1

Table 3. The chemical properties of the cements.

Chemical Properties	SO3, %	Cl−, %	LOI, %	Insoluble Residue, %	Na2O, %	K2O,%	Total Alkali, % (Na2O Equivalent)
CEMI	2.86	0.0342	2.74	1.02	0.37	0.56	0.74
CEMII	2.73	0.0326	-	-	0.62	0.59	1
CEMIII	2.29	0.0166	1.68	0.75	0.25	0.55	0.6
CEMIV	2.56	0.031	-	-	-	-	-

(LOI: Loss on ignition).

Table 4. The particle size distribution of the CEN standard sand.

Sieve Size (mm)	2	1.6	1	0.5	0.16	0.08
Total Residue on Sieve (%)	0 ± 5	7 ± 5	33 ± 5	67 ± 5	87 ± 5	99 ± 1

Table 5. The properties of the used superplasticizer.

Properties	Homogeneity	Chemical Structure	Form	Color	Density at 20 °C, g/cm3	Solid Content, %	pH Value	Chloride Content, %
Results	Homogeneous	Polycarboxylate Ether (PCE)	Liquid	Dark Brown	1.1337	28.04	5.06	0.0605

CEN standard sand consisting of round grains with a silica content of at least 98% was used for mortar preparation. The particle size distribution of the CEN standard sand is given in the Table 4. Commercially available Sikament RMC 1841 type superplasticizer was used in different ratios. The properties of the superplasticizer are given in Table 5.

2.2 Mix Proportion

Reference mortars (CEMI-0.0, CEMII-0.0, CEMIII-0.0, and CEMIV-0.0 without superplasticizer) and mortars with different superplasticizer ratios were prepared. The amount of water in the mortars was kept constant. The mixture designs of all prepared mortars are given in Table 6. Labeling was done according to the cement type and the percentage of superplasticizer. Specimens prepared with 0.8%, 1.0% and 1.2% superplasticizer were labeled as 0.8, 1.0, and 1.2 after the name of the cement type, respectively. Mortars were mixed in a mechanical mixer conforming to TS EN 196-1 [30] and the samples were poured into 40 mm × 40 mm × 160mm prismatic molds for strength tests. After 24 hours, the samples were demolded and then cured in lime-saturated water. For each combination, three specimens of 2, 7, and 28-days ages were prepared for the flexural strength and the compressive strength testing. Fresh samples were subjected to the flow table test to measure their consistency.

Table 6. Mixing proportion of the mortars.

Label	Cement (g)	Water (g)	Sand (g)	Superplasticizer (g)
CEMI-0.0 CEMII-0.0 CEMIII-0.0 CEMIV-0.0	450	225	1350	-
CEMI-0.8 CEMII-0.8 CEMIII-0.8 CEMIV-0.8	450	225	1350	3.6
CEMI-1.0 CEMII-1.0 CEMIII-1.0 CEMIV-1.0	450	225	1350	4.5
CEMI-1.2 CEMII-1.2 CEMIII-1.2 CEMIV-1.2	450	225	1350	5.4

2.3 Flow Table Test

The flow table test is a commonly used method for determining the consistency of freshly mixed mortars. The fresh state behavior of the mortars was characterized using the flow table test. This test was conducted in accordance with TS EN 1015-3 [31]. A brass cone was placed at the center of the flow table. The cone was filled with mortars in two layers and the layers were compacted 25 times with the tamper. The flow table was dropped 15 times, then the spread diameter of the mortar was measured.

2.4 Flexural Strength & Compressive Strength Tests

The flexural and compressive strength tests were performed according to the TS EN 196-1 standard [30]. Mortars were prepared and cast in 40 × 40 × 160 mm^3 prisms. The samples were cured in water until the test age and then tested for flexural and compressive strength on the 2nd, 7th and 28th days. After each prism was tested under flexural load, the two parts of the broken specimen were used for the compression tests. Pieces less than 65 mm in length were not tested under compression.

3 The research findings and discussion

3.1 Flow Table Test Findings

The flow table test was carried out to control the consistency of the different cement types produced from same clinker and containing different ratios of superplasticizer. The change in flow diameter of mortars with superplasticizer addition of is given in Figure 2.

The test results revealed that the fluidity of the mortars prepared with cements from the same clinker and the same w/c ratio is mainly controlled by the fineness of the cements. While more spreading was observed in CEMI and CEMII cements with less fineness, less spreading was observed in the finer CEMIII and CEMIV cements. These results were in line with the fact reported by Bonen and Sarkar [32] that pastes made with relatively low-fine cements had greater fluidity than high-fine cements for the same water/cement ratio. With an increase in fineness, the fresh state characteristics of the mixtures were adversely impacted due to the greater requirement for water to surrond the increased surface area of the finer cement particles.

When cement is mixed with water, particles of cement encapsulate water molecules and form a flocculent structure, resulting in a reduction in free water content. [33]. Therefore, the fluidity of the mortar mix decreased. The use of the superplasticizer increased the fluidity of the mortars.

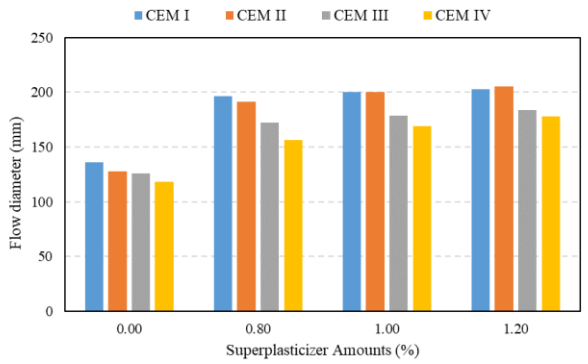


Figure 2. Flow diameter change of mortar with superplasticizer addition.

As the percentage of superplasticizer increased, the flow diameter also increased. Polycarboxylate ether (PCE) based superplasticizers consist of backbone chains with negative charges and side chains. The PCE-based superplasticizer's ability to increase the spread is based on its adsorption to the cement grain and dispersion of the particles by steric hindrance (Figure 3.). Increase in cement fineness used in the mixes reduced the effectiveness of the superplasticizer and resulted in an increase in the superplasticizer dosage required to achieve the given workability. These findings are similar to those reported by Bjornstrom and Chandra [34].

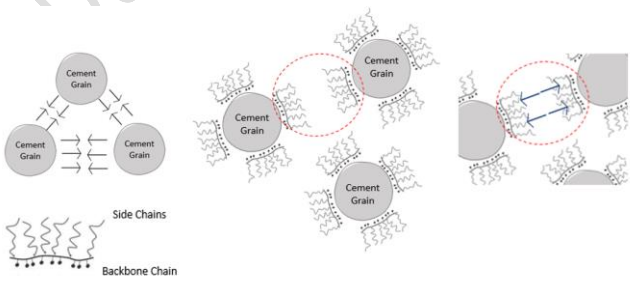


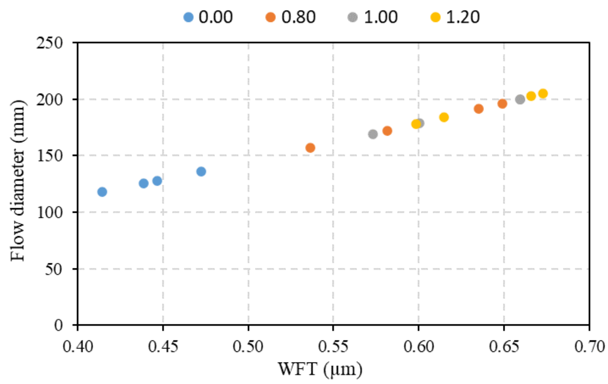
Figure 3. Superplasticizer working mechanism.

The thickness of the water film is a crucial factor that governs the flowability of fresh mortar. [35]. Various studies [36,37] reported that thickness of the water film is one of the most significant factors controlling fluidity of cement-based materials. Zhang et al. [38] proposed a relationship between the flow diameters of the mortar under different PCE dosages and the WFT as follows:

$$FD = 225.024 \text{ x } (WFT + 0.275)^{1.729} \quad (1)$$

Where FD is the flow diameter (in mm) and WFT is the water film thickness of the mortar (in μm). The proposed equation (Eq. 1.) was applied to examine the changes in the WFT of the mortars. Figure 4. illustrates the variation of WFT depending on the flow diameters and the amount of superplasticizer.

According to the results, flow diameter increased with the increase of WFT regardless of the cement type. CEMIII and CEMIV cement with finer particle sizes than CEMI and CEMII had a smaller WFT. This is consistent with the results of Kwan and Li [39] who asserted that the larger surface area of cementitious materials may result in a smaller WFT. While the amount of water was kept constant, the increase in the amount of superplasticizer caused an increase in the water film thickness (Figure 5). Due to the addition of superplasticizer, the flocculation tendency of the cement particles becomes difficult and the water film thickens [40]. As noted by Li and Kwan [41], a larger water film thickness reduced the shear stress required for the mixture to flow. In order to examine the effect of superplasticizer addition, WFT versus superplasticizer amount graph was drawn and regression analysis was performed to derive the best-fit curve describing the relationship. The equation and R^2 values of the obtained curves are presented on the Fig. 4b. Obtained R^2 values (≥0.94) prove that the WFT exhibits a linear variation depending on the amount of superplasticizer. However, it should be noted that the obtained equations and established relationships are specific to the mixing ratios and superplasticizer type used in the study. With the addition of superplasticizer, an increment of 40% to %50 in the WFT of mortars was observed. The highest increase was obtained in CEMII cement. This can be attributed to the lower surface area of the CEMII cement as well as its better compatibility with the admixture.



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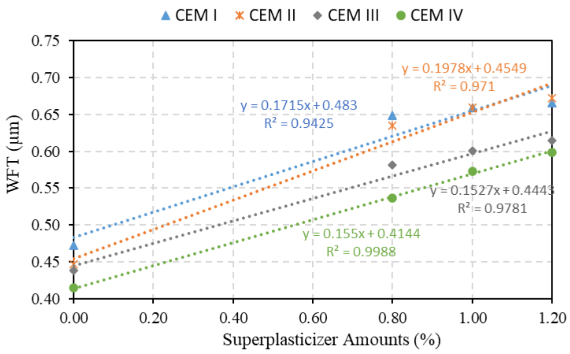


(b)

Figure 4. WFT variations (a) due to flow diameter, (b) due to superplasticizer amount.

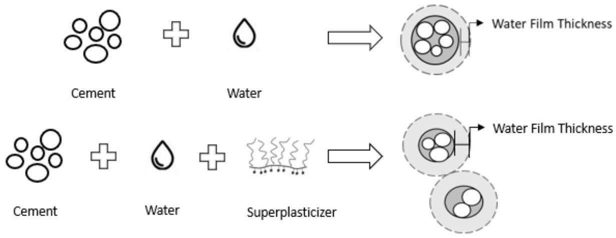
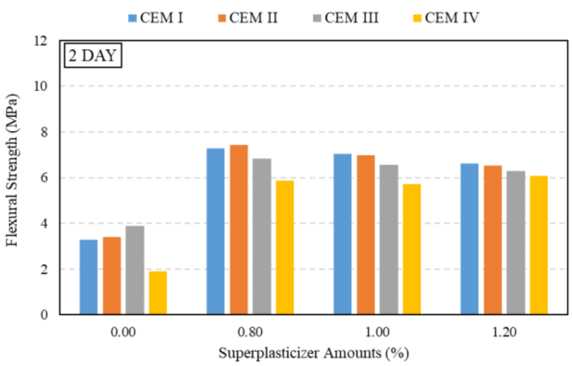


Figure 5. Water Film Thickness (Adapted from [40]).

3.2 Flexural Strength Test Findings

The flexural tests were performed on the specimens prepared of mortars with different dosages of superplasticizers. Figure 6 presents the impact of various superplasticizer dosages on the flexural strength.



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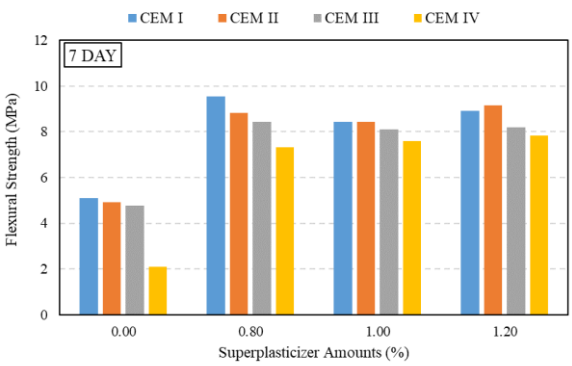

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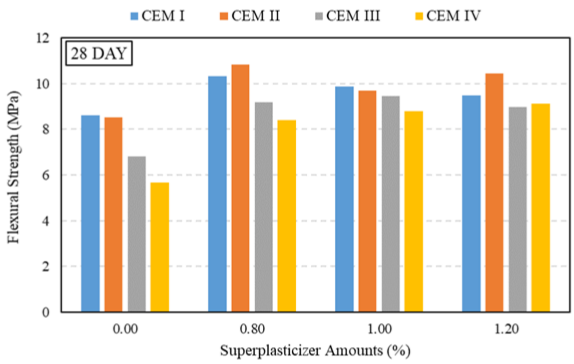


(c)

Figure 6. The effect of superplasticizer dosage on the flexural strength (a) 2 day, (b) 7 day, (c)28 day.

When the flexural strength test results were evaluated, it was determined that early strength (2 and 7 days) of the mortars prepared with CEMI, CEMII and CEMIII cement with the same strength classes were quite close to each other. The strength of the mortar designed with CEMIV cement, which has a lower strength class, was about half of the others. After 28 days, flexural strength of the mortar prepared with CEMIV cement increased significantly and approached that of the mortar with CEMIII cement. Mortars prepared with CEMI and CEMII cement achieved the highest flexural strength of about 8.5 MPa.

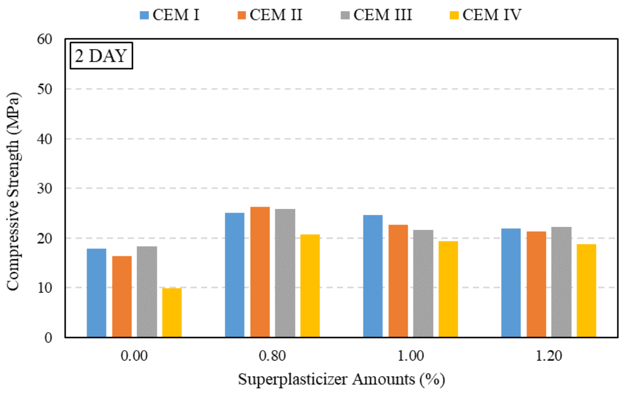
Due to the addition of superplasticizer, an increase in early strength of CEMI, CEMII, and CEMIII cements up to 2 times occurred. This increase was more pronounced in CEMIV cement, where it reached up to 3 times. The degree of this improvement in flexural strength at an early age depends on the superplasticizer dosage. When the 28-day flexural strength results were examined, it was observed that the efficiency of the superplasticizer decreased. With the addition of the superplasticizer, the difference between the flexural strengths of different cement types decreased, and the flexural strengths were close to each other.

3.3 Compressive Strength Test Findings

The compressive strength of mortars changes with time, and the mixture constituents have a significant effect on it. Therefore, compressive strength tests of mortars prepared using four distinct cement types derived from the same clinker were conducted at various ages. Figure 7 provides the effect of superplasticizer addition on the compressive strength of the mortars at different ages.

Considering the results, more than 50% of the 28-day compressive strength was achieved in the first days for all mixtures. As the clinker content decreased, the compressive strength decreased as well. The cement strength class had a significant effect on the strength of the mortar. At any age, CEMI cement, with the highest clinker content and a strength class of 42.5 R, exhibits approximately two times higher strength than CEMIV cement, with a low clinker content and a strength class of 32.5 R.

Determining the appropriate dosage of superplasticizer in cement mortars heavily relies on understanding its impact on compressive strength. Therefore, the compressive strengths of mortars containing varying superplasticizer contents were evaluated at different ages and compared with the reference mortar (Figure 7). All other variables were kept constant to see how the superplasticizer dosage would affect the results due to aging. Since the amount of water remains constant, the strength is expected to decrease as more water remains in the system with addition of superplasticizers. However, the increase in the strength of the mortars reveals that the water amount is insufficient for admixture-free mixtures. With the addition of superplasticizer, the workability of the mixtures increased, they compacted better, and their strength enhanced. The greatest contribution of superplasticizer addition to compressive strength was observed in the 7-day strength results. When the superplasticizer dosage was increased from 0% to 0.8%, the compressive strengths increased from 24.7 MPa to 44.1 MPa, from 24.7 MPa to 40.0 MPa, from 22.5 MPa to 37.9 MPa and from 10.5 MPa to 29.8 MPa for mortars containing CEMI, CEMII, CEMIII, and CEMIV, respectively.



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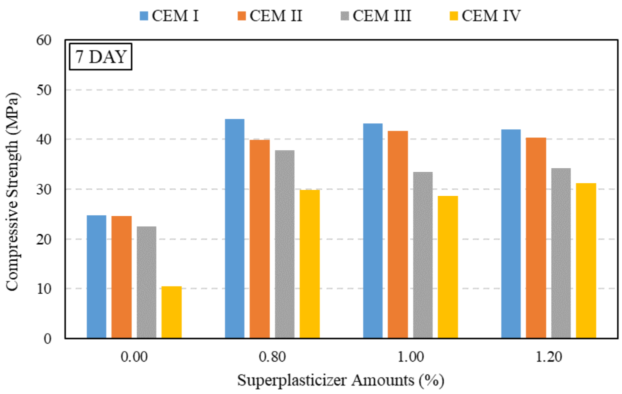

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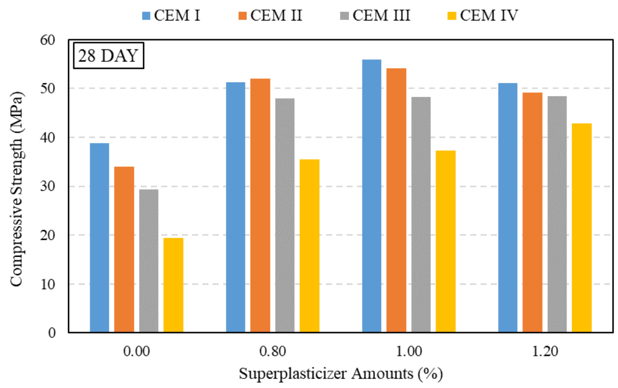

(c)

Figure 7. The effect of superplasticizer dosage on compressive strength (a) 2 day, (b) 7 day, (c)28 day.

With the addition of the superplasticizer, the early and final compressive strength increased significantly. This finding is consistent with that of other researchers [42,43]. The increment in compressive strength resulting from the inclusion of the superplasticizer is ascribed to the enhanced compaction effectiveness and the generation of a more compact mortar [44,45]. Increasing the amount of superplasticizer slightly decreased the level of improvement in early strength. Up to a specific limit, the compressive strength improved with the increment of the superplasticizer dosage, and beyond this dosage, the strength began to decrease. This limit was expressed as the optimum superplasticizers dosage. Upon evaluating the 28-day compressive strength, it was determined that the optimal dosage of superplasticizers for CEMI and CEMII cements is 1.0%, while for CEMIII and CEMIV cements, the optimal dosage is 1.2%.

4 Conclusions

The present study examined the fresh & hardened properties of CEMI, CEMII, CEMIII and CEMIV cement produced from the same clinker, and investigated the effect of adding superplasticizers at 0.8%, 1.0%, and 1.2% dosages to the related cements. The main conclusions are listed below.

- Experimental results showed that the flowability of the mortars prepared with cements produced from the same clinker and the same w/c ratio was controlled by the fineness of the cements. Regardless of the cement type used in the study, the increase in the fineness of the cement led to a decrease in its flowability, thus negatively affecting workability.
- With the addition of superplasticizers, the flow diameters of the mortars increased by 40-50% due to the dispersive effect of the additive on the agglomerated cement particles.
- According to the equation used in the study, the calculated water film thickness decreased linearly with the increase in the amount of superplasticizer in all mortar samples. Nevertheless, it would be beneficial to support the explanations based on numerically calculated water film thicknesses with experimental measurements.
- The inclusions of superplasticizers increased both the compressive and flexural strengths due to the reduction in the number of voids and the production of denser mortar through more effective compaction.
- Strength increased at a decreasing rate with increasing superplasticizer dosage, and beyond the saturation superplasticizer dosage, strength decreased with increasing dosage.
- The utilization of superplasticizers resulted in an enhancement of the compressive strength of the mortars. Nonetheless, the extent of this increase depended on the superplasticizers' dosage. Even though an increase was observed at different dosages, the effect was maximum at optimum dosage.
- The optimum dosage was determined as 1% for CEMI and CEMII and 1.2% for CEMIII and CEMIV. Increasing the dosage of superplasticizers above the optimum rate resulted in a decrease in compressive strength.
- While different cements will have different responses to a given admixture, different admixture

formulations will have different effects on a given cement. Therefore, further investigation could be done to understand the effects of different types of superplasticizers.

5 Author contribution statements

In this study, Writer 1 contributed to the formation of the idea, assessment of obtained results, spelling, and checking the article in terms of content; Writer 2 contributed to the literature review, data evaluation and article writing; Writers 3 and 4 contributed to the experimental studies and data collection.

6 Ethics committee approval and conflict of interest statement

The prepared article does not require approval of the ethics committee. In this article, there is no conflict of interest with any person or any institution.

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