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Determination of Compatible Plasticizer to Calcined Marl Blended Cement

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Abstract

Compatibility research between calcined marl (CM) blended cement and three plasticizers with different origins such as naphthalene sulphonate–formaldehyde (NSF), vinyl copolymer (VCP) and polycarboxylate ether (PCE) were performed. The effects of considered plasticizers on the workabilities and strengths of mortars containing calcined marl blended cements were investigated. Calcined marl needs such a research due to it is a relatively novel compared to other supplementary cementitious materials avalilable on the market. The blended cements in the study were obtained by partly replacement (0%, 10%, 30% and 50%) of calcined marl to clinker. Plasticizers used in the test mixtures were added with ratios such as 0.8% (low), 1.2% (medium) and 1.5% (high) to the mixing water. Density, slump-spread, ultrasonic pulce velocity (UPV) and strength on mortar samples were determined. According to the parameters examined in the study, the compressive strengths of mortars containing calcined marl blended cement improved up to 30% replacement ratio. And, the optimum compatibility between plasticizers and blended cements with calcined marl was determined as polycarboxylate ether (PCE) plasticizer with up to 1.2% usage ratio.

Keywords: calcined marl, blended cement, plasticizer, compatibility

Kalsine Marn Katkılı Çimentoya Uygun Akışkanlaştırıcının Belirlenmesi

Öz

Kalsine marn (CM) katkılı çimento ile naftalin sülfonat-formaldehit (NSF), vinil kopolimer (VCP) ve polikarboksilat eter (PCE) gibi farklı kökenli üç akışkanlaştırıcı arasındaki uyumluluk araştırması yapılmıştır. Dikkate alınan akışkanlaştıcıların kalsine marn katkılı çimento içeren harçların işlenebilirlikleri ve dayanımları üzerindeki etkileri incelenmiştir. Kalsine marn, piyasada bulunan diğer tamamlayıcı çimentolu malzemelere kıyasla nispeten yeni olması nedeniyle böyle bir araştırmaya ihtiyaç duymaktadır. Çalışmadaki katkılı çimentolar, kalsine marnın klinkere kısmen (%0, %10, %30 ve %50) yerdeğiştirilmesi ile elde edilmişlerdir. Deney karışımlarında kullanılan akışkanlaştırıcılar, karıştırma suyuna %0,8 (düşük), %1,2 (orta) ve %1,5 (yüksek) gibi oranlarda eklenmiştir. Harç numunelerinde yoğunluk, çökme-yayılma, ultrasonik darbe hızı (UPV) ve dayanımları belirlendi. Çalışmada incelenen parametrelere göre, kalsine marn katkılı çimento içeren harçların basınç dayanımları %30 yerdeğiştirme oranına kadar iyileşmiştir. Ayrıca, kalsine marn katkılı çimentolar ile akışkanlaştırıcılar arasındaki optimum uyumun %1,2' ye kadar kullanım oranına sahip polikarboksilat eter (PCE) akışkanlaştırıcısı olduğu belirlenmiştir.

Anahtar Kelimeler: kalsine marn, katkılı çimento, akışkanlaştırıcı, uyumluluk

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Introduction

Cement is a building material that needs to efficiency usage of energy and to improvement as environmental in terms of production techniques. To produce a ton of cement requires approximately 80 to 100 kWh of electrical power. Also during its production processes release average 0.95 tons of CO2 (Bildirici & Ersin, 2024). The current targets of the cement industry are to reduce these values. The easiest and most effective way to meet this need is to produce blended cement using high ratios of clinker substitute material. Thus, due to less clinker use, energy savings and low CO2 emissions action targets could be achieved. As seen, the cement industry has a global impact on CO2 emissions (Andrew, 2018; Xiang et al, 2024; Liu et al, 2024). Cement is also an important product that is closely related to the economic development of countries. Lately, many countries of the world have increasingly been produced blended cements with lower clinker ratios. (Guo et al., 2024). Naturally, the high replacement demand in blended cement brings plasticizer demand. In this case, compatibility between plasticizer and blended cement is new problem that needs to be solved. Also, one of the important problems in these applications is the fewness of research of compatible plasticizer to mineral additive type used for blended cements. As known, the solution of this compatibility problem is generally neglected in the literature studies. As also seen from studies, (El Bitouri et al., 2022; Elistratkin et al., 2019; Kermani et al., 2024; Kulshreshtha et al., 2023; Nitin et al., 2024) cement-plasticizer incompatibility is caused by both plasticizer-origin and cement-origin factors. For this reason, researches on especially blended cements and compatible plasticizers to its should be carried out together. The manufacturers of plasticizer industry should design products by focusing to experimental studies and considering all the factors in terms of compatibility.

Fly ash, furnace slag, and silica fume are additive materials for blended cements. These materials will always be needed in cement production due to their environmental impact, energy saving and performance improvement aims. However, even today cement factories already consume approximately 90% of the available blast furnace slag (Moukannaa et al., 2020). These current materials cannot meet increasingly demand in the future. Therefore, recently, interest in calcined clays has been increasing (Tironi et al., 2013) Because, clays have proven to be abundantly available almost everywhere in the world. And, they show high pozzolanic activity when they are calcined at appropriate temperatures (Bahhou et al., 2024).

Natural marl is a material that contains clay and limestone in certain percentages (35-85% clay and 15-65% limestone) and is as called “clay contaminated” by limestone within the clay group (Bahhou et al., 2021). But, when it is calcined at appropriate temperature, it shows high pozzolanic activity. Also, calcined marl with high potential waiting to be discovered is a novel alternative natural SCM for Portland cement clinker that exhibits synergistic reactions thanks to its clay and limestone contents in its natural structure (Danner, 2018). To summarise, calcined marl offers a tremendous potential as SCM that can be replaced with cement clinker.

In previous studies, the blended cements containing calcined marl used with high replacement ratios performed quite well in terms of strength and durability (Danner et al, 2015; Ng &,Justnes, 2015; Rakhimova et al., 2021; Sposito et al., 2022). But the using of calcined marl significantly decreases the flow of the cement due to high absorption of water. Therefore, when calcined marl is used with high replacement, it should be used with a suitable plasticizer. It needs to study on the compatibility of plasticizers to be used with the cement. In the literature, the research on compatibility between plasticizer and calcined marl blended cements is very few. This is related to the fact that calcined marl is still novel and non-common SCM.

Through this study, it is aimed to obtain scientific data that will provide the commonly use with optimum values of local-natural marl as a novel SCM for blended cement productions. In the author's previous studies, pozzolanic activity of calcined marl, some properties of blended cements with calcined marl and mortars containing blended cements with calcined marl were extensively studied (Akgün, 2019; Akgün, 2020; Akgün, 2021). The scope of this study is to examine the compatibility between blended cements containing calcined marl and plasticizers. In this study, the physical property, compressive strength, ultrasonic pulse velocity (UPV) and slump-spread tests on the mortar samples produced with blended cements containing calcined marl and plasticizers with different orgin were performed. All results of test series were examined comparatively with each other and reference samples.

Materials and Methods

Plasticizers affect the physical and mechanical properties of cementitious systems. Many studies have been conducted on the compatibility of plasticizers with different cements. In these studies, it was concluded that cement–plasticizer compatibility depends on the change in the chemical and physical properties of the concrete components (Tunc, 2024; Kobya et al., 2024; Murugan et al., 2024). However, plasticizier-cement compatibility is still seen as an unknown. The objective of the study is investigated how compatibility between blended cements containing calcined marl and chosen plasticizers affects workabilities and strengths of produced samples.

The CEM I 42.5 R type Portland cement in accordance with EN 197-1 (CEN, 2012) standard was used as binder. The some properties of cement obtained by the manufacturer are given in Table 1.

Table 1. Properties of CEM I 42.5R.

Chemical Properties		Physical Properties		Clinker Components (%)	
Sulfur Trioxid (SO3) (%)	3.02	Specific Gravity (g/cm^3)	3.12	C_3S	54,78
Chloride (Cl^-) (%)	0.0252	Specific Surface (cm^2/g)	3210	C_2S	26,11
Loss on Ignition (%)	3.06	Initial Setting Time (min.)	170	C_3A	11,83
Insoluble Residue (%)	0.76	Volume Expansion (mm)	2.0	C_4AF	0.91
Mechanical Properties					
2 Days Compressive Strength (MPa)		32.30			
28 Days Compressive Strength (MPa)		53.00			

It was used natural calcined marl as replacement material with clinker in production of blended cement. Natural marl was obtained from Sinop/Erfelek, Black Sea Region of Türkiye. The total percentage of Si, Al and Fe oxides in the chemical content of natural marl is 76.43% by weight. Its specific surface area and density is 4630 cm2/g and 2.70 g/cm3, respectively. These properties indicate that natural marl has pozzolanic activity according to TS 25 (TSE, 2011). However, natural marl must be calcined at the optimum temperature for its pozzolanic activity. To calcine natural marl at the most suitable temperature, as stated in many technical literature if clay contain marl it should be calcined at least 800 °C (Tironi, 2013). Likewise, optimum calcination temperature of marl was determined by TGA method as 8000C in the author's previous study (Akgün, 2019). Therefore, natural marl was calcined at 800 °C. CEN standard sand defined with EN196-1 (CEN, 2016). was used as aggregate in production of samples. The specific grain size distribution CEN Standard sand ranges between 0.08 and 2.00 mm.The sieve residue in % of CEN standard sand for 2.00, 1.60, 1.00, 0.50, 0.16 and 0.08 squared mesh sizes in mm is 5.28, 33.82, 67.35, 86.83 and 99.71, respectively. The specific gravity and water absorption capacity of CEN standard sand were obtained as 2.64 and 0.6% by mass, respectively, in accordance with the EN 1097-6 (CEN, 2022) standard. The maximum moisture content is 0.2%. The sand is portioned in bags of 1350 (± 5) g. Three types of blended cements in three different replacement ratios (10%, 30% and 50%) were used in preparing mortar mixes. Chemical admixtures are used in practice for many purposes such as gaining higher strength by reducing the water/cement ratio, reducing the amount of cement in mass concrete to reduce the heat of hydration, or ensuring the same workability and easy settling (Plank et al, 2015). In this study, investigations were carried out on determination of compatible plasticizier to improve of parameters such as workability, physical property, ultrasonic pulse velocity and strength of test samples produced by blended cement with calcined marl and different plasticiziers. The three different plasticizers were added to the mixing water in proportion (0.8%, 1.2% and 1.5%) to the cement weight. According to American Concrete Institute (ACI), plasticizers could be classified in terms of chemical content as sulphonate melamine formaldehyde, sulphonate naphthalene formaldehyde, modified lignosulphonates, and copolymers contain sulphonic and carboxyl groups (ACI, 1987). The study was carried out comply with this classification. In tests, in order to determinate the effectiveness of mortar mixtures prepared with grinded natural marl, different plasticizers such as polycarboxylate ether (PCE), naphthalene formaldehyde sulfonate (NSF) and vinyl copolymer (VCP) were added to the mixing water. The selected plasticizers are brown in color, liquid form and comply with EN 934-2 (CEN, 2013). They were used by adding 0.8% (low), 1.2% (medium) and 1.5% (high). The specific gravity of the plasticiziers are 1.05 g/cm3, 1.12 g/cm3 and 1.14 g/cm3, respectively. All of tests were performed at the end of curing period (28days). The mortar samples without calcined marl and plasticizers were also cast as the comparison samples. The city mains water was used as mixing water. A view of calcined natural marl samples (a), standard sand (b) and plasticiziers (c) are seen in Figure 1.

Figure 1. A View of Calcined Natural Marl Samples (a), Standard Sand (b) and Plasticiziers (c)

SEM (Scanning Electron Microscope) investigations were carried out in Ordu University Central Research Laboratories to define the general surface morphologies of natural marl and marl samples calcined at 800oC. SEM examinations of natural marl and calcined marl samples with calcination process were carried out by grinding as finer than Portland cement fineness for pozzolanic activation purposes. In order for the samples to turn into conductive material as required by SEM examinations, the surface of the marl samples on the carbon band of the device called "sputter" was gold plated in standard sizes. In this study, natural marl calcined at 800°C was replaced in the ratios 0%, 10%, 30% and 50% with clinker. And then, mortar samples were produced with the obtained blended cements. In the study, mini slump-spread tests were carried out in accordance with (EN 12350-8) (CEN, 2019) to determine the compatibility between blended cement containing calcined marl and plasticizers in terms of workability. The high amount of slump-spread indicates that the mortar has a high ability to move under its own weight. In this regard, the greater the amount of slump-spread the greater the degree of workability. On the other hand, it was carried out physical property (EN 12390-7) (CEN, 2019), ultrasonic pulse velocity (ASTM C597-22) (ASTM, 2023) and compressive strength with (EN 196-1) (CEN, 2016) tests on mortar samples to determine the compatibility between cement containing calcined marl and plasticizers in terms of strength. The tests were carried out "Building and Material Laboratory" of Technical Sciences Vocational School in Ordu University. The samples were 28 days during the strength tests. The results obtained were compared with each other. The labels of test series are given in Table 2. The amounts of components included in the mortar mixtures are given in Table 3. Mortar samples were produced as a series of 40 tests with 50x50x50mm cube samples under laboratory conditions with a temperature of 20±2ºC and relative humidity of 60±5%. The samples were 28 days at the time of the tests. The tests were carried out at Ordu University, Vocational School of Technical Sciences, Construction and Materials Laboratory. Some images of production and tests are given in Figure 2.

Table 2. The Labels of Test Series

The labels of test series	Description
CM0	0% marl and 0.8%-1.2%-1.5% ratios of plasticiziers
CM0PCE0.8, 1.2, 1.5	PCE; Polycarboxylate Ether
CM0NSF0.8, 1.2, 1.5	NSF; Naphthalene Sulphonate–Formaldehyde
CM0VCP 0.8, 1.2, 1.5	VCP; Vinyl Co-Polymer
CM10	10% marl and 0.8%-1.2%-1.5% ratios of plasticiziers
CM10PCE0.8, 1.2, 1.5	PCE; Polycarboxylate Ether
CM10NSF0.8, 1.2, 1.5	NSF; Naphthalene Sulphonate–Formaldehyde
CM10VCP0.8, 1.2, 1.5	VCP; Vinyl Co-Polymer
CM30	30% marl and 0.8%-1.2%-1.5% ratios of plasticiziers
CM30PCE0.8, 1.2, 1.5	PCE; Polycarboxylate Ether
CM30NSF0.8, 1.2, 1.5	NSF; Naphthalene Sulphonate–Formaldehyde
CM30VCP0.8, 1.2, 1.5	VCP; Vinyl Co-Polymer
CM50	50% marl and 0.8%-1.2%-1.5% ratios of plasticiziers
CM50PCE0.8, 1.2, 1.5	PCE; Polycarboxylate Ether
CM50NSF0.8, 1.2, 1.5	NSF; Naphthalene Sulphonate–Formaldehyde
CM50VCP0.8, 1.2, 1.5	VCP; Vinyl Co-Polymer

Table 3. The Amounts of Components in the Mortar Mixtures

Components (kg/m^3)	M0	M10	M30	M50
Portland Cement	350	315	245	175
Calcined Marl	-	35	105	175
Water	175	175	175	175
Water/Cement	0.50	0.50	0.50	0.50
Standard Sand	1350	1350	1350	1350
Plasticizier ratios (%)				
0%	-	-	-	-
0.8%	2.80	2.80	2.80	2.80
1.2%	4.20	4.20	4.20	4.20
1.5%	5.25	5.25	5.25	5.25

Figure 2. A View for Some of Production and Test Methods

Results and Discussions

The findings obtained from the study on the compatibility of different plasticizers to blended cements containing calcined marl are outlined below.

SEM photographs showing the general surface morphology for natural marl and calcined marl samples are given in Figure 3. It could seen internal micro structure, grain and void structure of natural and calcined marl samples from SEM photos. The amorphous structures of natural and calcined marl samples and the changes in grain structure after calcination are observed.

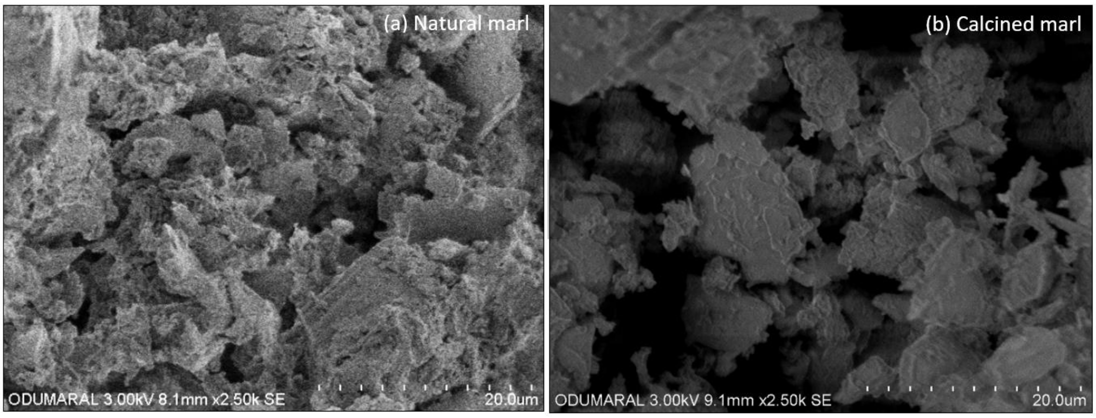

Figure 3. SEM Photos of Natural (a) and Calcined (b) Marl Samples

The variation graphs of average densities (Figure 4), slump (Figure 5), spread values (Figure 6), ultrasonic pulse velocities (Figure 7) and compressive strengths (Figure 8) for replacement, type of plasticizer and ratios of mortars containing calcined marl blended cement are given below.

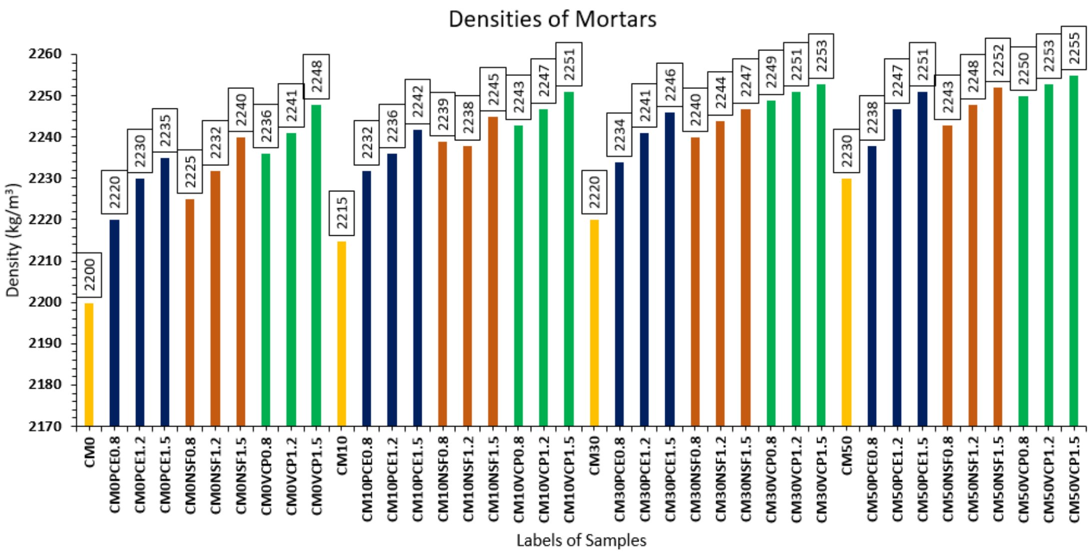

Figure 4. Densities of Mortar Samples

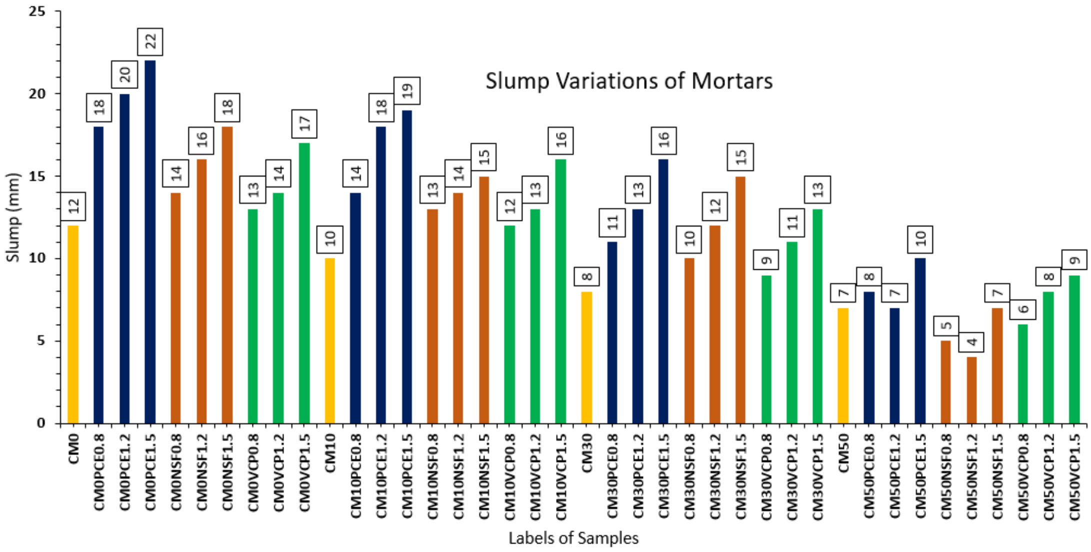

Figure 5. Slump Variations of Mortar Samples

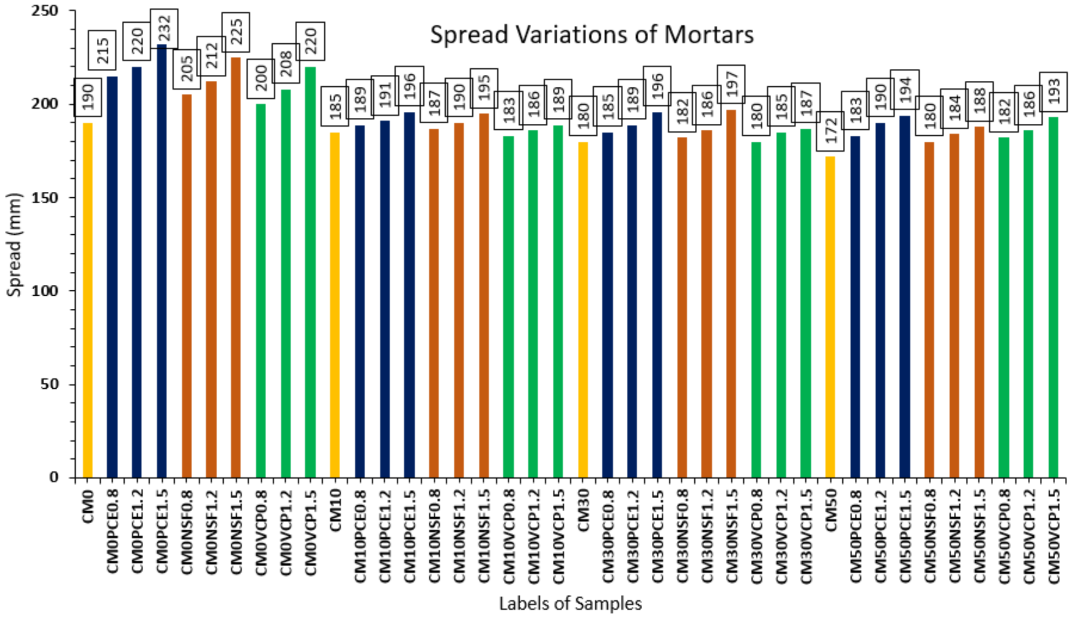

Figure 6. Spread Values of Mortar Samples

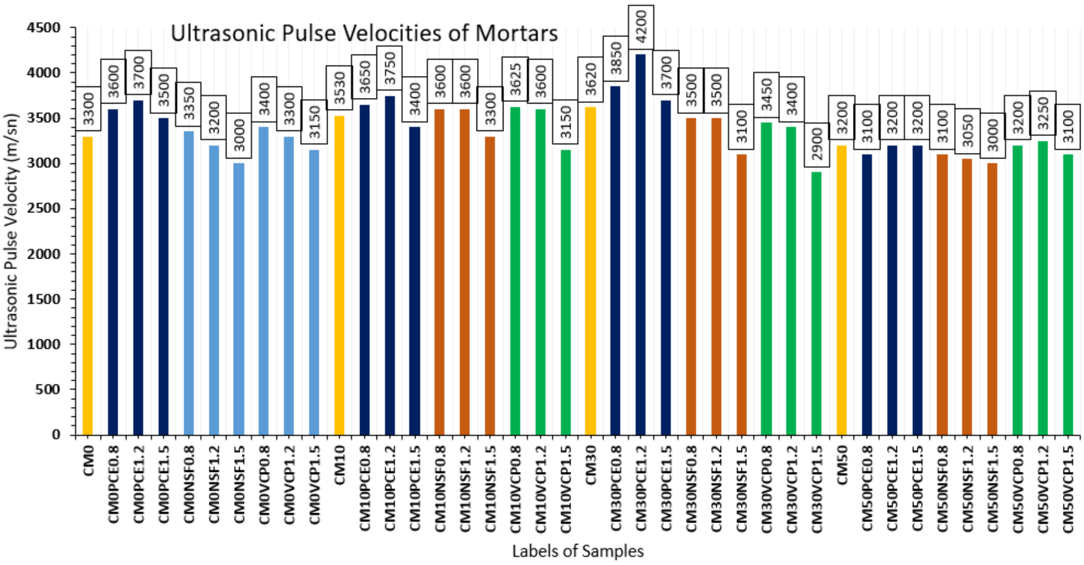

Figure 7. Ultrasonic Pulse Velocities of Mortar Samples

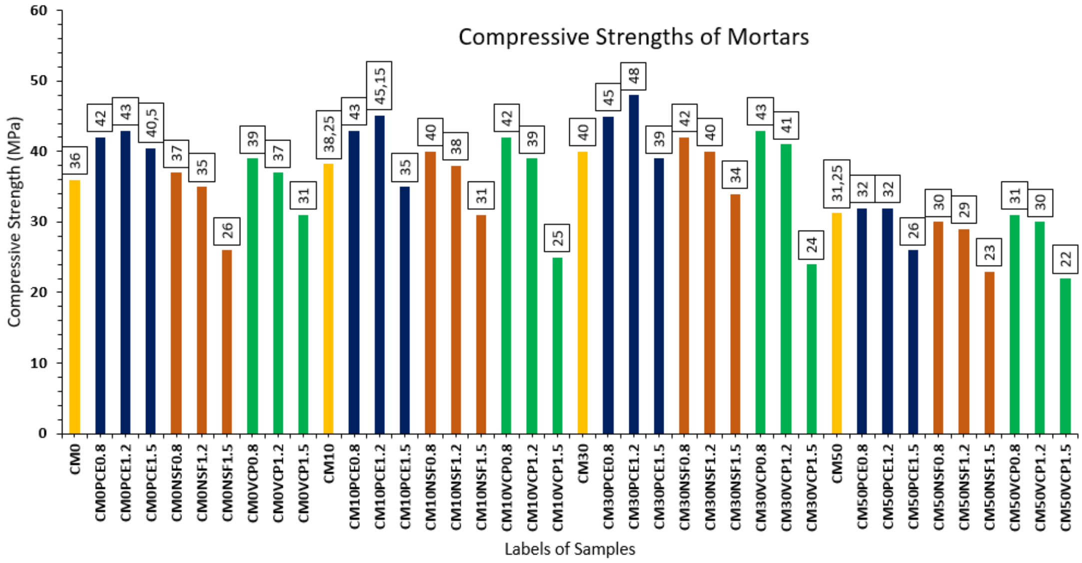

Figure 8. Compressive Strengths of Mortar Samples

According to variation graphs of densities, the variations in the densities of the samples in the series (CM0, CM10, CM30 and CM50) with calcined marl replacement but without plasticizer increase as the amount of calcined marl replacement increases. This is a result of the voidless internal structure due to the filler effect caused by the mineral additive being ground below the cement fineness.

According to the slump-spread values of mortar samples, it was observed that the slump-spread values in all of mortar mixtures decreased with increase in calcined marl replacement. And, as the plasticizer usage ratio increased, the slump-spread values increased. Among the plasticizers used in the study, PCE plasticizer is the most compatible plasticizer with calcined marl blended cements in terms of workability.

All test program series were compared in terms of only strength improvements. It was more higher 11.11% in those without plasticizer (for CM30), 19.44% in those without calcined marl but with plasticizers (for CM0PCE1.2) and 33.33% in those with both calcined marl and plasticizer (for CM30PCE1.2) than strengths in CM0 series. According to these ratios, it is thought that the use of plasticizers in designs using mineral additives improves the pore structure by providing regular distribution of the mixture and also improves the final product strength (Cho et al., 2005; Plank et al., 2008). The variations in densities support this mechanism related to the development of compressive strength.

In this study, the optimum calcined marl replacement ratio was determined as 30%. Some strength decreases were observed in replacement ratio of 50% calcined marl. However, the strengths in the study were determined on 28-days samples. Therefore, there is also an expectation that the late-age strengths of the samples will improve with the late-age additional binders due to the pozzolanic activity of the calcined marl used in the mixtures (Danner et al., 2012).

The compressive strengths of samples were compared in terms of type of plasticizer and usage ratio. It is seen that the most suitable plasticizer is PCE in terms of higher compressive strengths for both the series without marl and the series containing NS and MP plasticizers. These increases in strength are more apparent for the 0.8% and 1.2% ratios. This is because water bleeding and segregation were observed during the production of mortars containing 1.5% PCE and calcined marl.

On the other hand, the UPVs of samples were compared. The compressive strength and UPVs of the samples show similar tendency. The internal structural variations of mortar samples support the compressive strength test results. This expected situation from literature studies is an indication that the production of test samples was carried out in accordance with the standard conditions. It also shows that the determination method with the UPV device which is one of the non-destructive testing methods for determining the quality of concrete can be used to provide data for the uniaxial compressive strength determination method which is one of the destructive testing methods.

Conclusions and Recommendations

The conclusions obtained from findings of the study on the compatibility of different plasticizers to blended cements containing calcined marl are,

- The density, slump-spread, UPV and compressive strengths of mortar samples produced by using calcined marl and CEM I 42.5R type cement showed variations according to the origin of used plasticizers.
- According to the results and discussions, it was determined that polycarboxylate-based (PCE) plasticizer was more effective than NS and MP plasticizers under the conditions of this study in terms of compatibility to blended cement containing calcined marl. Literature supports the compatibility of PCE plasticizer for the dispersion of calcined clay blended systems (Ng & Justnes, 2015; Li et al, 2021).
- The test results carried out on mortar samples containing calcined marl blended cement in terms of workability and strength showed that PCE is the plasticizer that compatible with calcined marl blended cements up to 30% replacement ratio and its usage ratio is up to 1.2%. Because, it was observed water bleeding and segregation during the production of mortars added PCE at 1.5% ratio.
- Here, it should be emphasized that cement-plasticizer interaction is important in terms of fresh concrete properties and strength, therefore cement-plasticizer compatibility tests should be carried out before application.

Author Contribution

The authors co-wrote, read and approved the manuscript.

Ethics

There are no ethical issues regarding the publication of this article.

Conflict of Interest

The authors declare that they have no conflict of interest.

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