



LIMESTONE AND NATURAL POZZOLAN BLENDED CEMENTS: EVALUATING SULFATE RESISTANCE FOR SUSTAINABLE CONSTRUCTION

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Highlights

- Blended Cements for Sustainability: Limestone and pozzolan cements as alternatives.
- Sulfate Resistance Comparison: Limestone cements resist sulfate better than pozzolan.
- Long-Term Durability: Blended cements last 360 days under sulfate exposure.

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ABSTRACT: This study aims to compare the behavior of ordinary Portland cement, sulfate resistant cement, natural pozzolan blended cement and limestone blended cement, produced in the same strength class as Portland clinker, under the influence of sulfate. For this purpose, five different cements were produced in Konya Cement production facilities. Characterization and sulfate tests were carried out on the cements. The strengths of the cements blended with limestone and natural pozzolan were determined in different sulfate environments and under normal curing conditions, and their behavior under sulfate effect was observed during a 360-day monitoring period. The experimental results revealed that the sulfate resistant cement with the lowest C₃A content had the highest sulfate resistance. Moreover, the limestone blended cement showed a superior performance compared to the natural pozzolan blended cement. The compressive strength of the natural pozzolana blended cement was 50.1 MPa at 28 days and decreased by about 6% to 47.1 MPa after 360 days of sulfate exposure. On the other hand, the 28-day compressive strength of the limestone blended cement was 49.8 MPa, while it remained almost unchanged at 50 MPa after 360 days of sulfate exposure. These results show that limestone and natural pozzolan blended cements have the potential for widespread use in construction applications in line with environmental sustainability goals.

Keywords: Blended Cements, Limestone, Natural Pozzolan, Sulfate Resistance

1. INTRODUCTION

Concrete, a material that is utilized extensively across various construction projects and architectural endeavors on a global scale, is recognized as one of the most prevalent building substances in existence. Notably, beyond the numerous advantages that concrete possesses, which include remarkable compressive strength, cost-effectiveness, and the versatility to be molded into an array of desired shapes and forms, it is also imperative to acknowledge that it is not devoid of certain inherent disadvantages. Concrete elements must be durable to extreme environmental conditions. Various measures are taken to ensure the durability of concrete structures, especially in environments rich in aggressive chemicals (acid, base, salt, etc.). With external protection, aggressive chemicals can be prevented from reaching concrete structures, but this is not possible in every case. In addition to external protection, the durability of the elements can be increased by optimizing the concrete or cement components. Factors such as soil, groundwater, and seawater that can come into contact with various concrete elements can contain high levels of sulfate. The incorporation of cement replacement materials (CRM) such as fly ash, blast furnace slag, calcined clay, and natural pozzolans in concrete mixtures can enhance the longevity of concrete components, especially their resistance to sulfate attack [1], [2], [3], [4]. Moreover, the use of SCMs in mixtures does not only provide durability, but also helps to reduce the maintenance and repair needs and the carbon footprint of the cement and concrete industry by reducing the amount of clinker. In addition to traditional CRMs, additives such as anti-sulfate inhibitors, hydrophobic agents, various waste powders, nanomaterials, etc. are the subject of various research in the literature to increase the impermeability and sulfate resistance of concrete [5], [6], [7].

With increasing environmental concerns, there is a growing interest in using recycled materials [8],

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reducing greenhouse gas emissions in cement production and reducing the clinker/cement ratio. Various academic and industry studies on topics such as limestone calcined clay cement (LC3), blended cement and geopolymer concrete are progressing with determination [9], [10], [11], [12], [13]. The most important consideration in these studies, which support global sustainability initiatives, should be both maintaining performance standards and minimizing environmental impact. Binders with reduced clinker to cement ratios are candidates to become widespread in traditional construction applications and replace ordinary Portland cement. However, a comparative study of the behavior of blended cements produced with various mineral additives that meet certain performance standards under aggressive conditions is required. This is essential to facilitate the selection of cements for long-term applications and to ensure that they meet the demands of modern construction practices.

Considering the environmental impacts, various innovation efforts in the cement and concrete industry are continuing to increase rapidly. Recently, studies have been carried out on the production of cement with different contents without compromising performance. Reducing the amount of clinker, which is responsible for high greenhouse gas emissions, per unit of cement is the basis of these studies [14]. In studies where cement or clinker is substituted with various mineral additives, the main priority is to reduce the amount of cement as much as possible without losing performance. Cement and/or concrete production methods that show the same performance compared to the reference sample and have a lower carbon footprint are considered acceptable within the scope of emission reduction studies. Although specimens substituted with various additives show sufficient strength in the short term, various durability studies are also needed. While reducing clinker content can reduce carbon emissions, it can also affect the performance of concrete if not managed properly [15]. In particular, substituting clinker or cement with materials such as limestone and natural pozzolana can improve durability parameters such as freeze-thaw, alkaline silica reaction, chloride resistance as well as environmental advantages [16], [17], [18]. However, it is important to design the mix carefully to avoid problems such as low early strength and workability. In addition, although it can be inferred from life cycle analysis studies in the literature which cement is more environmentally friendly, it is worth investigating which cement performs better against specific durability problems. This study is based on investigating the sulfate resistance of cements containing different proportions of clinker but belonging to the same class.

The aim of this study is to compare the sulfate resistance of limestone blended, natural pozzolan blended and ordinary cements produced with Portland clinker of the same strength class. For regions containing sulphate groundwater and regions with sulphate content in the soil structure, it would be appropriate to use a sustainable cement type with easy accessibility, high sulphate resistance and low cost. For this reason, it was decided to investigate the effect of limestone blended cements with low fineness and high Blaine values on sulfate resistance with the hypothesis that the filling effect of limestone will increase the filling effect and impermeability. Various properties of the blended and ordinary cements produced were defined and their behavior under the effect of sulfate was compared.

2. MATERIAL AND METHODS

In this study, five different cements were produced to investigate the behavior of different cements under sulfate effect. To be used in this study, ordinary Portland cement (CEM I 42.5), limestone (CEM II/A/L 42.5), natural pozzolan (CEM II/A/P 42.5) and limestone-natural pozzolan blended (CEM II/A 42.5) cements and sulphate resistant cement (CEM I SR-5 42.5) were produced at Konya Cement production facilities. The additive ratios of the produced cements were determined considering the condition of belonging to the same strength class (42.5). The physical properties, compressive strengths and performance of the cements produced within the scope of the study were examined after sulfate effect.

2.1. Material

2.1.1. Clinker substitutes

In Central Anatolia and especially around Konya region, it is difficult to access additives such as blast furnace slag, fly ash, silica fume in terms of cost and sustainability. Due to the abundance of limestone and natural pozzolana resources in this region [19], limestone and natural pozzolana are more advantageous than other additives in terms of access and cost. Considering these issues, natural pozzolana and limestone were preferred as clinker replacement materials in this study. Detailed analysis of limestone (LS) and natural pozzolan (NP) used in the study is presented in Table 1.

Table 1. Analysis of limestone and natural pozzolan used as additives in cements produced

Chemical and physical content		Limestone	Natural pozzolan
SiO ₂	%	1.04	64.64
Al ₂ O ₃	%	0.42	16.93
Fe ₂ O ₃	%	0.11	5.33
CaO	%	53.93	4.64
MgO	%	0.30	1.08
SO ₃	%	0.09	0.00
K ₂ O	%	0.02	3.42
Na ₂ O	%	0.02	3.00
Cl	%	0.018	0.002
Loss of ignition	%	44.67	2.64
Moisture	%	1.86	13.0
Pozzolanic activity	MPa		5.7
Reactive SiO ₂	%		28.1

2.1.2. Cement composition

The compositions and 28-day compressive strengths of different cements produced in Konya Cement production facilities within the scope of this study are presented in Table 2. All cements produced are in the same class (42.5) according to TS EN 197-1.

Table 2. Cement compositions and 28-day compressive strengths

Sample	Short name	Clinker (%)	Gypsum (%)	Natural Pozzolan (%)	Limestone (%)	Comp.Str. (MPa)
CEM II/A M (P-L) 42.5 R	CEM II/A	78.05	5.89	8.70	7.36	47.0
CEM I 42.5 R	CEM I	88.13	5.14	0.0	6.44	46.0
CEM I 42.5 R SR-5	SR-5	88.75	4.42	0.0	6.43	48.6
CEM II/A (L) 42.5 R	CEM II A/L	77.21	5.89	0.0	14.64	46.7
CEM II/A (P) 42.5 R	CEM II A/P	78.56	5.11	15.56	0.77	45.1

2.2. Method

2.2.1. Determination of physical, chemical and mechanical properties of cement

The cements produced within the scope of this study were tested for insoluble residue (IR) according to TS EN 196-2, Blaine surface area and sieve residue according to TS EN 196-6, normal consistency and setting time according to TS EN 196-3, elemental composition by XRF and compressive strength on days 2, 7 and 28 according to TS EN 196-1. With these analyzes, the basic parameters that are decisive in the

identification of cement were determined

2.2.2. Preparation of mortar mixtures and tests

Standard mortar mixtures were prepared in accordance with TS EN 196-1 with the cements produced within the scope of this study and whose basic properties were defined. The prepared mixtures were placed in 40x40x160 mm molds and kept in the mold for 24 hours. After 24 hours, the specimens were removed from the mold and cured in lime-saturated water until the test day.

The compressive strengths of the hardened mortar specimens cured in lime-saturated water were determined at 28, 40, 90, 150 and 360 days of age. In addition, standard mortar mixtures were prepared from the cements produced to be used in sulfate resistance tests. After 28 days of normal curing, the age of the specimens was considered zero and they were placed in 3% magnesium sulfate (MgSO_4) solution. The compressive strengths of the specimens kept in sulfate solution were similarly determined at 28, 40, 90, 150 and 360 days of age and compared with the specimens kept in normal curing. The work flow diagram of the study is presented in Figure 1 and photos of some of the prepared and tested samples are presented in Figure 2.

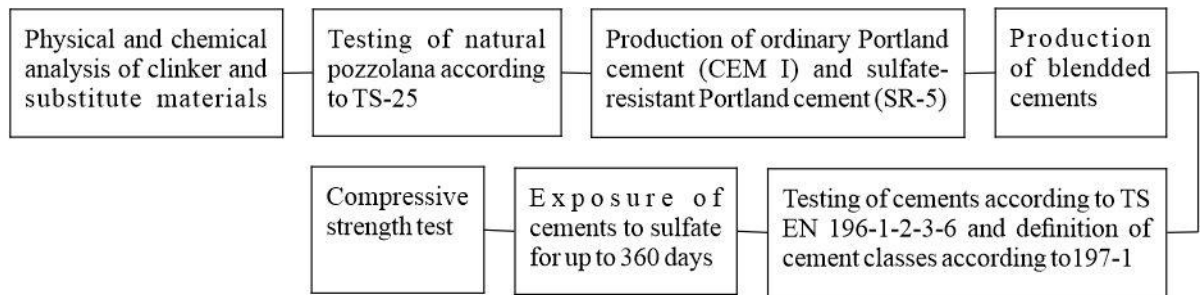


Figure 1. The flow chart

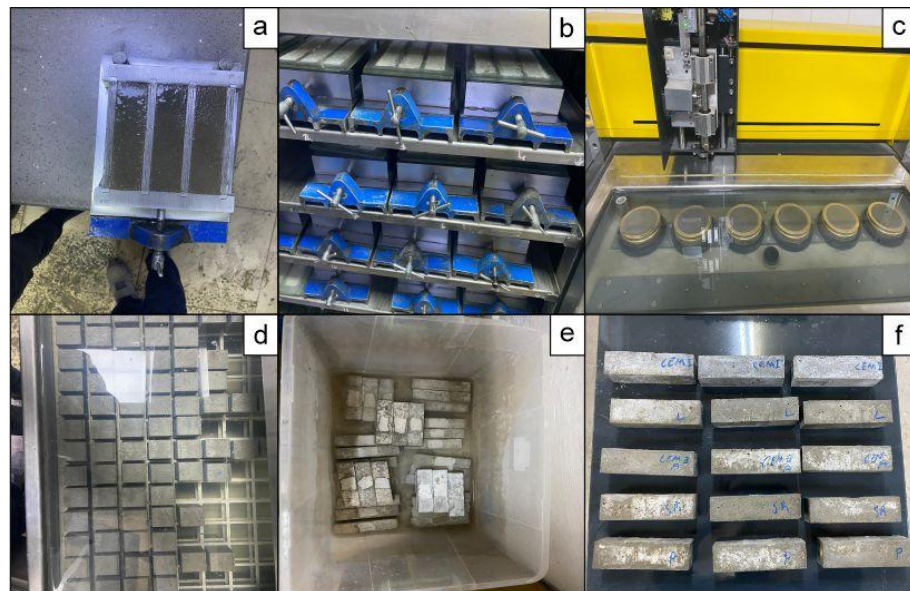


Figure 2. Molded fresh mortar (a), specimens kept in curing cabinet (b,d), setting time determination test (c), samples kept in MgSO_4 solution (e,f)

3. RESULTS AND DISCUSSION

3.1. Physical, chemical and mechanical properties of cements

The chemical and physical properties of the produced cements are presented in Table 3 and Table 4, respectively. According to the chemical analysis results, the lowest amount of C_3A was found in SR-5 cement and the highest amount of C_3A was found in CEM II A/P cement.

Table 3. Chemical composition of cement

Content (%)	CEM II/A M (P-L) 42.5 R	CEM I 42.5 R	CEM I 42.5 R SR-5	CEM II/A (L) 42.5 R	CEM II/A (P) 42.5 R
SiO ₂	21.26	18.6	19.38	17.97	24.86
Al ₂ O ₃	5.12	4.52	4.35	4.38	6.13
Fe ₂ O ₃	4.44	4.16	4.54	3.96	5.51
CaO	58.52	61.36	61.71	62.34	53.61
MgO	1.06	1.1	1.15	0.97	1.18
SO ₃	3.55	3.21	2.89	3.55	3.2
K ₂ O	0.88	0.69	0.61	0.74	1.03
Na ₂ O	0.49	0.33	0.32	0.36	0.63
Cl	0.0177	0.00203	0.0182	0.0179	0.0165
C ₃ A	6.06	4.94	3.85	4.91	6.92
C ₄ AF	13.51	12.66	13.82	12.05	16.77

Table 4. Physical properties of cement

Sample	CEM II/A M (P-L) 42.5 R	CEM I 42.5 R	CEM I 42.5 R SR-5	CEM II/A (L) 42.5 R	CEM II/A (P) 42.5 R
Bf (cm ² /g)	4228	3721	3689	4713	4046
32 µm (%)	6.9	-	-	5.5	9.2
45 µm (%)	2.5	5.6	3.0	1.7	4.1
IR (%)	7.68	0.45	0.54	2.14	13.58
Standard consistency (%)	29.0	27.8	28.3	28.3	29.3
Initial set (min.)	140	150	155	150	255
Final set (min.)	220	220	230	220	345

When the Blaine fineness and sieve residue values of the cements were analyzed, the cement with the highest Blaine fineness was found to be CEM II/A/L. The cement with the lowest Blaine fineness is SR-5. In addition, the 32 µm sieve residue of LS-blended cement is 5.5%. Both sieve residue and Blaine fineness values indicate that LS-blended cement is the most finely ground type among the cements to be compared. The standard consistency of the cement types containing NP is slightly higher than the other cement types. When the physical and chemical analyzes were examined, it was seen that all cement types prepared for use in the experiment fulfilled the requirements of TS EN 197-1 standard.

The 2,7 and 28-day strength results of the cements used in the study are given in Figure 3. All cement types used belong to 42.5 MPa strength class. In addition, it was determined that the 2-day early strength values, which is a requirement of TS EN 197-1 standard, were above 20 MPa in all cement types.

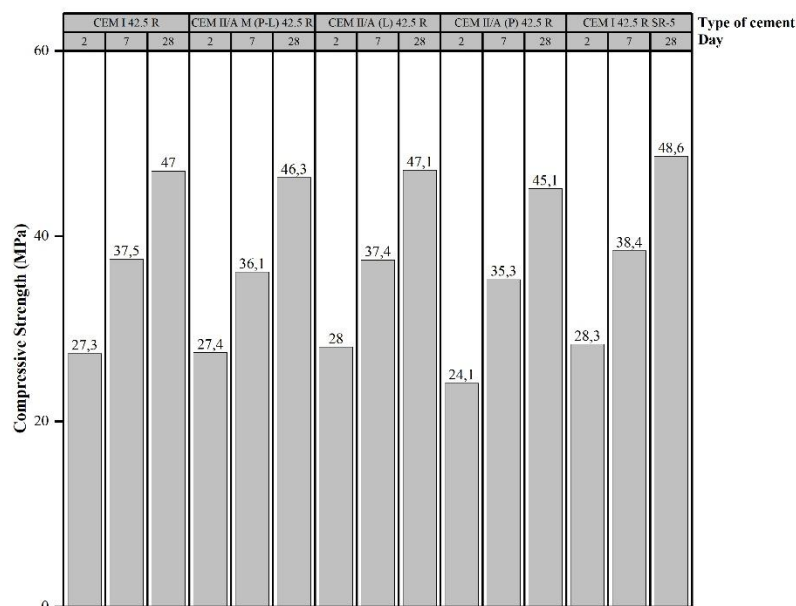


Figure 3. Compressive strength results of cements

All cements belong to the same strength class. Replacing clinker with various additives can often result in strength reduction, depending on pozzolanic activity and substitution rate [20]. However, the component ratios and Blaine fineness of the cements were optimized in this study so that the cements belonged to the same strength class. SR-5 cement (88.75%) with the highest clinker content has the lowest Blaine fineness, while CEM II A/L cement (77.21%) with the lowest clinker content has the highest Blaine fineness. It is known that cement in which some of the clinker has been replaced with inert LS would need to be ground finer to perform as well as cement with higher clinker content [21]. However, not only the fineness but also the type and amounts of components affect the strength [22]. Compared to other cements, the setting time of NP-blended cement was slightly delayed and the early strength was slightly lower. This is related to the slow progression of pozzolanic reactions and their effectiveness at later ages. The better early strength and setting time of cement with LS and NP (CEM II/A) compared to cement with NP alone (CEM II A/P) may be related to the fineness. The strength and setting time results of CEM II/A cement, which contains less pozzolanic material but has higher Blaine fineness, showed that the cement fineness can prevent pozzolanic reactions at early age. In addition, the pozzolanic activity of the natural pozzolan used in the experiment was determined to be 5.7 MPa and the reactive SiO_2 content was determined to be 28.1% according to TS-25. Although the natural pozzolan used meets TS-25 requirements, the reactive silica content is low.

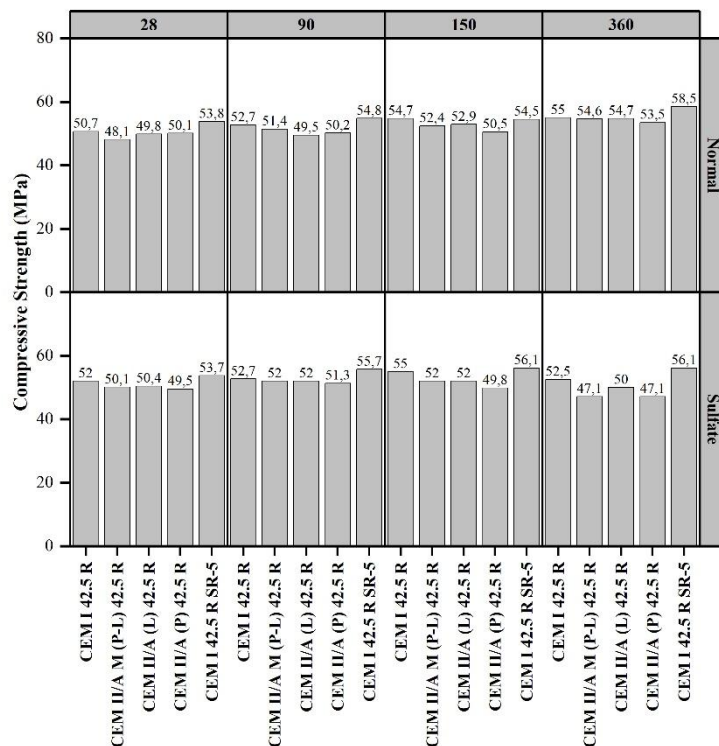
According to TS EN 197-1 and TS-25, the limits that Portland cements should meet and the information showing the properties of the cements produced within the scope of the study are presented in Table 5.

3.2. Sulfate tests

After determining that the cements produced in the first stage were in compliance with the standards and belonged to the same strength class, 120 samples were prepared for each cement type in the second stage and 60 of them were kept in magnesium sulfate solution and 60 of them were kept in a standard curing pool. The compressive strengths of the specimens kept under different curing conditions were determined at 28, 90, 150, and 360 days. The compressive strength results of the mortar mixtures prepared with the cements used in the experiment are presented in Figure 4.

Table 5. Limits of TS EN 197-1 and TS-25

Limits of TS EN 197-1						Cement within the scope of this study				
Cement type	Early strength (2-days MPa)	Strength (28-days MPa)	SO ₃ (%)	C ₃ A (%)	Initial setting (min.)	Early strength (2-days MPa)	Strength (28-days MPa)	SO ₃ (%)	C ₃ A (%)	Initial setting (min.)
CEM I 42.5 R	≥ 20	62.5 ≥ X ≥ 42.5	≤ 4	-	≥ 60	27.3	47	3.21	4.94	150
CEM I 42.5-R-SR5	≥ 20	62.5 ≥ X ≥ 42.5	≤ 4	< 5	≥ 60	28.3	48.6	2.89	3.85	155
CEM II/A (L) 42.5 R	≥ 20	62.5 ≥ X ≥ 42.5	≤ 4	-	≥ 60	28	47.1	3.55	4.91	150
CEM II/A (P) 42.5 R	≥ 20	62.5 ≥ X ≥ 42.5	≤ 4	-	≥ 60	24.1	45.0	3.2	6.92	255
CEM II/A M (P-L) 42.5 R	≥ 20	62.5 ≥ X ≥ 42.5	≤ 4	-	≥ 60	27.4	46.3	3.55	6.06	140
Limits of TS 25						Pozzolan within the scope of this study				
Type	Reactive silica (%)	SiO ₂ +Al ₂ O ₃ +Fe ₂ O ₃ (%)	SO ₃ (%)	Cl (%)	Pozzolanic activity (MPa)	Reactive silica	SiO ₂ +Al ₂ O ₃ +Fe ₂ O ₃	SO ₃ (%)	Cl (%)	Pozzolanic activity (Mpa)
Natural pozzolan	≥ 25	≥ 70	≤ 3	≤ 0.1	≥ 4	28.1	81.57	0	0.002	5.7

**Figure 4.** Compressive strength results of cements in sulphate and normal curing

All tests were carried out after a normal curing period of 28 days, assuming the specimen age to be zero. According to the test results, an increase in strength was observed for all cement types up to day 150, including the specimens in sulfate solution. An increase in strength is expected in the initial periods. The reason why the strength decrease could not be detected within 150 days is that magnesium sulfate reacts very slowly with the calcium-silica-hydrate gels that provide the binding of the cement paste. This reaction causes some of the C-S-H to dissolve.

At the end of 360 days, compressive strength results showed that the strength of the specimens in sulfate solution decreased in all cement types. It was observed that the strength increase of the specimens kept in magnesium sulfate solution stopped and even decreased. The reason for the strength decrease is that magnesium sulfate causes the formation of gypsum in the concrete and the formation of ettringite of the metastable C_4ASH_{12} products. In addition, magnesium sulfate causes some of the C-S-H gels to dissolve. The silica gel formed as a result of this reaction reacts very slowly with magnesium hydroxide and causes the formation of crystalline magnesium silicate with no binding value [23], [24]. These reactions are the main reasons for the strength decrease.

One of the most important parameters affecting the sulfate resistance of concrete to external sulfate attack is the amount of tricalcium aluminate (C_3A) in the cement. Low C_3A content can result in higher sulfate resistance by limiting the formation and transformation of ettringite [25], [26]. This research shows that SR-5 cement with the lowest C_3A content has the highest sulfate resistance. No loss of strength was observed in SR-5 cement maintained in a sulfate environment for 360 days. In general, mixtures prepared with CRM-substituted cements show improved durability performance in aggressive environments such as sulfate [27], [28]. In this study, NP blend cement showed the lowest sulfate resistance, with strength decreasing after 90 days. One of the reasons for the improved durability of CRM-substituted cements is the lower amount of C_3A in diluted clinker [29]. In this study, NP blend cement had the highest C_3A content. Therefore, the performance is usually worse than the other cements. The other types of cement have a similar C_3A content to ordinary Portland cement (CEM II/A has a higher C_3A) and showed almost similar sulfate resistance. Physical factors such as a dense matrix and low porosity due to low water/binder content can also limit sulfate penetration and improve durability [30], [31]. Limestone is known to aid in the formation of a dense matrix and limit the formation of gypsum and ettringite [32]. However, it should be noted that a high degree of limestone substitution can increase porosity. In limestone mixing systems, the formation of stable hydration products requires less sulfate compared to ordinary Portland cement, which can increase the sulfate resistance of the mix [33]. However, it is also reported in the literature that limestone substitution of 20% or more has a negative effect on sulfate resistance [32]. As a result, when fineness and content ratios are optimized, blended cements produced with less clinker can show at least as much sulfate resistance as ordinary Portland cement. Moreover, LS-blended cement can outperform NP-blended cement.

Baldermann et al. (2018) concluded that when limestone powder is substituted with cement at ratios less than 50%, it can show better sulfate resistance than ordinary Portland cement due to the reduction in total porosity and pore diameter [34]. Makhloufi et al. (2016) revealed that the substitution of limestone and natural pozzolan with cement has a positive effect on sulfate resistance [35]. In the present study, similar results were obtained to the studies reported in the literature and the performances of the additives were compared with each other.

4. CONCLUSIONS

The following conclusions were made in this research carried out with different cements of the same strength class.

Cements with different clinker contents were ground at different Blaine finenesses to provide the same strength class according to TS EN 197-1. In this way, cement containing approximately 88% clinker and cement containing approximately 77% clinker can belong to the same strength class. It is understood that the clinker difference of about 10% can be compensated by increasing the Blaine fineness.

The setting time of NP blended cement was delayed compared to other cements. However, an improvement in setting time was found when LS admixture was used together with NP. Similar situation was observed in both early and ultimate strength. When NP blended cement was compared with NP-LS blended cement, NP-LS blended cement gave better results. However, the difference in Blaine fineness should not be ignored. The pozzolanic activity of the NP used in the study was determined as 5.7 MPa and the reactive SiO_2 ratio was 28.1%. Although the NP used met the requirements of TS 25, it has low reactivity. It can be concluded that finer ground inert limestone can perform better than the NP with

pozzolanic activity of 5.7 MPa.

The experimental study demonstrated the sulfate resistance of limestone and natural pozzolan blended cements. Due to its low C3A content, SR-5 cement did not lose strength for 360 days in sulfate solution.

Compared to natural pozzolan blended cement, limestone blended cement has higher sulfate resistance. NP blended cement suffered a strength loss of approximately 6% after 360 days of sulfate exposure, while LS blended cement did not show any strength difference. This may again be related to Blaine fineness. It was concluded that the dense microstructure and low void ratio of the finer grinded LS blended cement made sulfate penetration more difficult.

The results show that blended cements containing less clinker can be as durable as conventional Portland cement when appropriate fineness and content ratios are selected. This study emphasizes that limestone and natural pozzolan blended cements can contribute to environmental sustainability goals.

Credit Authorship Contribution Statement

[Ahmet Yiğit] Conceptualization, Methodology, Investigation, Resources, Formal Analysis, Writing - Original Draft; [Furkan Türk] Conceptualization, Methodology, Writing - Review & Editing, Visualization; [Ülkü Sultan Keskin] Conceptualization, Methodology, Resources, Supervision, Funding Acquisition, Project Administration, Validation, Writing - Review & Editing.

Data Availability

The data presented in this study are fully contained in the figures and tables of this article.

Declaration of Ethical Standards

The authors declare that the study complies with all applicable laws and regulations and meets ethical standards.

Declaration of Competing Interest

One of the authors of this study works in the cement factory where the production takes place. Authors declare that there is no conflict of interest that this affects the results of the study.

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