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Research Article

Mechanical and microstructural properties of mortars: Obsidian powder effect

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ABSTRACT

Concrete has been the world's most produced and utilized building material for years due to its economic and easy accessibility. However, it attracts attention due to the CO_2 emitted from cement, the raw material of concrete, during the production and consumption stages. Although there are different research studies to reduce this emission, one of the most logical solutions is to use pozzolanic materials with cement and reduce the need for cement. This paper investigated the general material characteristics of mortar samples generated by substituting obsidian powder with pozzolanic properties into cement at different ratios by weight. Mortar specimens with varying proportions of obsidian, such as 0%, 10%, 20%, and 30% by weight, were subjected to mechanical tests at 3, 7, 14, and 28 days. Material tests like X-ray diffraction (XRD) and scanning electron microscopy (SEM) were employed to characterize the material. As a result of the mechanical tests, 42.52 MPa compressive strength was obtained from the 28-day reference sample, while 44.331 MPa compressive strength was obtained from the mortar sample with 30% obsidian substitution. The outcomes of this paper noted that obsidian powder, which has pozzolanic properties, increased the mechanical strength of cementitious mortar specimens. This work indicates the suitability of using obsidian as pozzolan material with cement was determined.

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1. INTRODUCTION

If we have to list the materials that are most produced, used, and studied in the civil engineering discipline, it is clear that concrete is the most common material after water [1]. Concrete production, which is increasing with each passing day because it is cheap and durable, reached 32 billion tons in 2021 [2, 3]. Since traditional concrete cannot be produced without cement, cement production is increasing daily depending on concrete production. Today, as global warming and greenhouse gas effects have improved considerably, the impact of carbon footprints is gaining importance daily. In this context, CO_2 emissions from cement production and consumption constitute 7% of total emissions [4, 5]. Innovative technologies are needed to reduce cement production daily for a sustainable environment. Considering the advantages of traditional concrete produced with cement, one of the ideal solutions is to decrease the use of cement in the mix by using pozzolanic additives [6, 7]. Generally, materials with pozzolanic properties are known as not binders but gain binder properties when used with cement. Using pozzolanic materials in concrete production reduces the cost of cement and contributes to the durability properties of concrete, resulting in an ergonomic concrete output. Most importantly, it helps to reduce the amount of CO_2 emitted by diminishing the amount of cement produced.

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Table 1. Mixture calculations of the mortar series-produced

Mixture name	Binder (g)		Water (g)	CEN standard sand (g)	Water/ binder ratio
	Cement	Obsidian			
S1 (reference)	450	0	225	1350	0,5
S2	405	45	225	1350	0,5
S3	360	90	225	1350	0,5
S4	315	135	225	1350	0,5

There are many studies in which pozzolanic materials have been substituted with cement [8–12]. Ustabaş and Ömür [13] searched the changes in the strength and hydration heat of industrial waste by-products and obsidian powder that they substituted for cement. They stated that the strengths obtained from the samples produced by substituting 40% of pozzolanic materials such as obsidian, fly ash, and blast furnace slag into cement mortars were similar to those of unsubstituted cement mortars. Keçek et al. [14] examined the utilization of tuff as a mineral admixture in cement. They looked into the effect on strength values by substituting tuffs with different surface area values, which were ground at other times, into cement. The maximum strength of 37,45 MPa was obtained in 28 days in substituted cements. Zhao et al. [15] conducted a study to measure the pozzolanic activity of waste clay brick powders with different specific surface areas. They discovered that the strength values grew with the rise in surface area. Çullu et al. [16] used volcanic rocks as a substitute for cement at different ratios between 10%–50% to determine the pozzolanic properties of various types of volcanic rocks. They found that the ideal mixing ratio was 10%. Araújo et al. [17] observed an 11% higher increase in compressive strength compared to cement samples when the ceramic waste used as a cement substitute was 25% by weight. Aruntaş et al. [18] focused on the differences created by substituting blast furnace slag and hydrated lime into cement-based mortars and pastes. Although hydrated lime and blast furnace slag contributed to the concrete at some points within the scope of the study, it was observed that lower strength was obtained compared to the reference specimens.

A comprehensive literature review indicated that pozzolanic material experiments were conducted to determine the pozzolanic characteristics of waste and inert materials. Although there are various studies on common materials like fly ash, blast furnace slag, etc., there are few studies on innovative materials such as obsidian. This study investigates the substitution of obsidian, which has pozzolanic properties, into cement. Compressive strength, XRD, and SEM-EDS analysis results of concrete mortars were investigated.

2. MATERIALS AND METHODS

Within the scope of the study, concrete mortars were produced by substituting 10%, 20%, and 30% obsidian to cement. The obsidian rocks collected from nature were first crushed in a jaw crusher and pulverized by grinding in a ball mill. The material/ball weight ratio in the ball mill was 1/24. The information and mixing parameters of the mortar series used in the study are given in Table 1. The mortar specimens were cast according to ASTM C109 [19]. After casting, the mortars were left in the molds for 24 hours before being removed and water-cured in the curing pool.

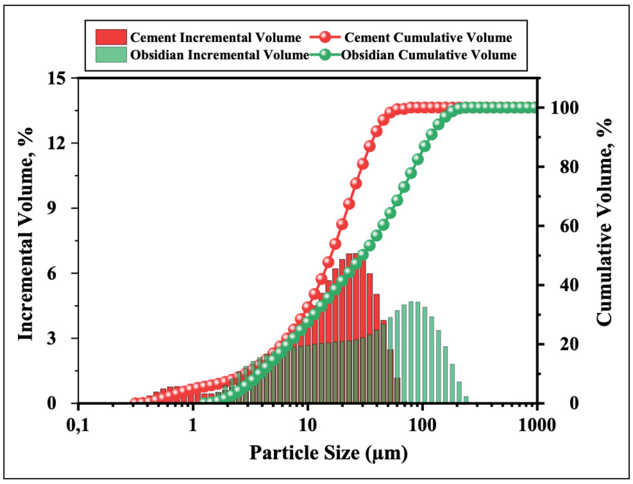

Figure 1. Cumulative volume and incremental volume granulometry curves of obsidian and cement.

2.1. Components

2.1.1. Obsidian

Obsidian rocks obtained from the Çağırankaya region of İkizdere District of Rize Province, Türkiye, were used in the study. The specific gravity of the ground obsidian powders is 2.6, and the average grain size is 53.786 µm. According to ASTM C618 [20], obsidian powder has N class natural pozzolanic properties as $SiO_2+Al_2O_3+Fe_2O_3$ >70, CaO<18, SO_3<4 and L.O.I<10. The granulometer curve of obsidian is given in Figure 1. The chemical contents obtained from XRF analysis of obsidian are given in Table 2.

2.1.2. Cement

The Portland cement type is CEM II/A-L 42.5 R, obtained from Trabzon Aşkale, which suits TS EN 197-1:2012 [21] requirements. The cement's specific gravity is 3.15 gr/cm^3, with an average particle size of 35.452 µm. The granulometer curve of the cement is given in Figure 1. Table 2 shows the chemical contents determined from XRF analysis of the materials employed as binders in the study. Also, the chemical phase structure of cement is depicted by XRD in Figure 2.

2.2. Methodology

In this study, The objective was to explore mortars' mechanical and microstructural qualities by inserting obsidian, which possesses pozzolanic capabilities, into cement at 10%, 20%, and 30% by weight. Mortar cube specimens of 50x50x50 mm^3 were cast in line with ASTM C109 [18], with four separate batches created using two different binders. For the mortars subjected to mechanical tests on other days, such as 3, 7, 14, and 28, 3 samples were poured in each series, and three compressive strengths were averaged. The experiments were carried out at Recep Tayyip Erdoğan University Building Materials Laboratory, and Figure 3 depicts a flow chart covering the working processes.

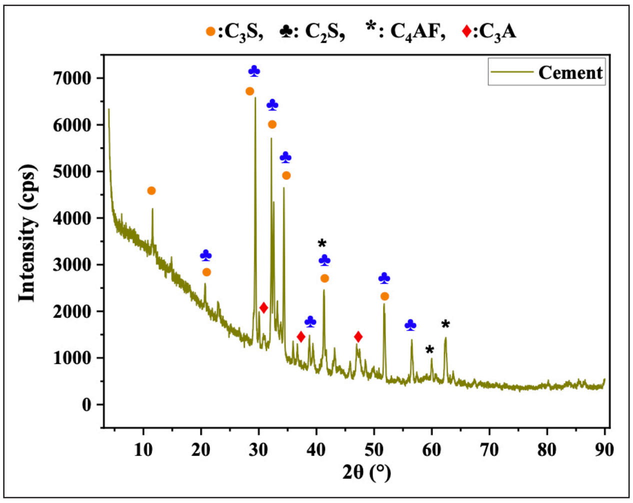

Figure 2. XRD pattern of the cement.

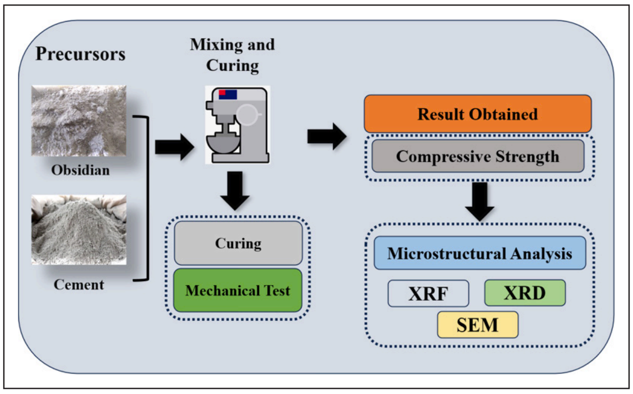

Figure 3. Flowchart of laboratory and test processes.

2.2.1. Mechanical Strength Experiments

The compressive tests were done on mortar specimens of dimensions (50x50x50 mm^3) previously cast and exposed to water curing on various days. All conditions, such as laboratory conditions, mixing water temperature, materials used, mechanical tests, etc., were implemented by requirement of ASTM C109 [18].

2.2.2. XRD Analysis

XRD analysis is one of the standard methods utilized to determine the characteristic mineral content of mortar samples. This study used a Rigaku SmartLab instrument (Cu-K-beta type PW1830, 2θ:4-90) for the analysis. The mortar samples were subjected to mechanical tests and ground in a ring grinder to prepare them for XRD analysis. 40 kV operating voltage, 0.02 step size, and 40 Ma emission current were selected as test parameters.

2.2.3. SEM Observations

The EOL JSM-6510 type SEM device was used to analyze the pores of mortar samples in which cement and obsidian were used as binders using a formal tone. The electron microscope has parameters like an accelerating voltage of 20 kV, a current emission of 0.9 nA, a magnification range of 5 to 1,000,000, and a resolution of 3 nm. To produce a clean image, the samples prepared for SEM examination were held at 50 °C for a specific time before vacuuming. The mortar samples were then put in the test equipment, covered with Au film, and examined under 50 to 10,000 magnification.

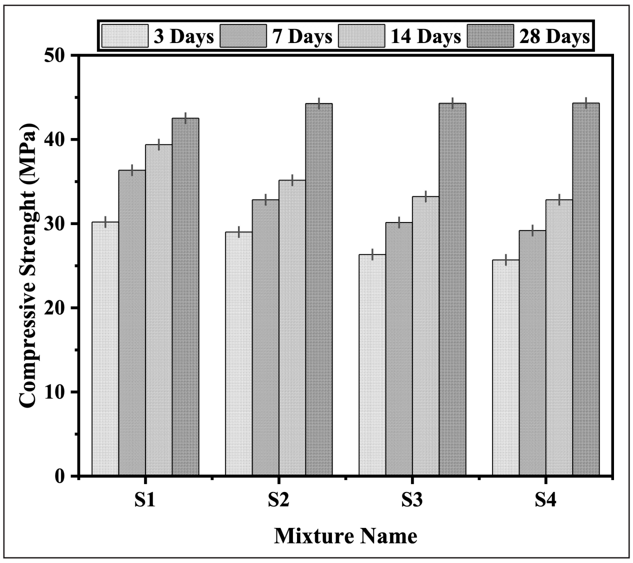

Figure 4. Compressive strength bar graphs of different mortar series for 3, 7, 14, and 28 days.

Table 2. Chemical composition of binders (%)

Chemical content	Obsidian	Cement
SiO_2	73,624	17,564
Al_2O_3	13,779	4,619
K_2O	5,296	1,006
Na_2O	3,959	–
Fe_2O_3	1,263	2,942
CaO	1,044	63,452
TiO_2	0,207	0,343
BaO	0,083	0,018
MgO	0,075	–
Mn_3O_4	0,055	0,051
SO_3	0,022	2,727
P_2O_5	0,02	0,118
SrO	0,018	0,023
L.O.I.	0,51	7,34

3. RESULTS AND DISCUSSION

3.1. Mechanical Strengths

Figure 4 demonstrates the compressive strengths of 50x50x50 mm mortar specimens generated by ASTM C109 at 3, 7, 14, and 28 days. Mechanical tests on mortar specimens were canceled if they exceeded the average value by more than ±10%. New specimens were cast to repeat the testing. Figure 4 illustrates that the maximum and lowest compressive strength values in 3 days were measured from the S1 and S4 series, respectively. At 3-day values, the compressive strength values of the S2, S3, and S4 series are 96%,

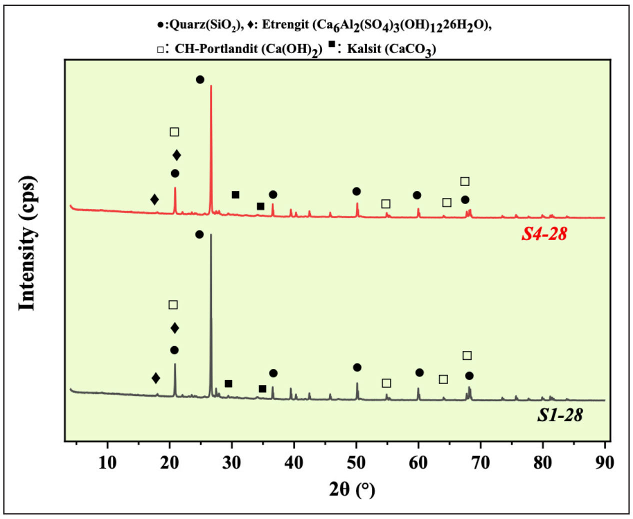

Figure 5. XRD patterns of S1 and S4 mortar samples.

87%, and 85%, respectively, compared to the strength of reference S1 which contains 100% cement. There is an inverse correlation between the amount of obsidian in the mixtures and the compressive strengths. The 7-day values show a similar trend to the 3-day compressive strengths. The compressive strength values of the S2, S3, and S4 series are 90%, 83%, and 80% respectively compared to the strength of S1. Obsidian content and compressive strength have an inverse relationship, similar to the 3-day values. However, it is seen that the proportional difference between the 7-day values and the reference sample increases. The 14-day strengths are identical to the three and 7-day strengths.

However, when 28-day strengths are analyzed, it is understood that there is a linear connection between obsidian content and compressive strength. The compressive strength values of the S2, S3, and S4 series are 104%, 104.1%, and 104.2% respectively compared to the strength of S1. When the research is evaluated according to TS EN 197-1 [21], the early compressive strength of the mortar specimens (7 days) should be more than the standard's requirement of 16 MPa, and the 28-day standard compressive strength should be more significant than 42.5 MPa. The 7-day and 28-day strengths of the reference control series S1 were 36,35 MPa and 42,52 MPa, respectively. When the other mixture series are considered, it is seen that the other series also have compressive strengths higher than the values required by the standard. Generally, the compressive strength of the obsidian-containing mortar specimens was lower than the reference specimen in the early age period but exceeded the reference specimen at 28 days. This demonstrates that obsidian advantages affect mortar specimens' compressive strength during the standard age period. Obsidian's favorable influence on compressive strength during the typical age period is assumed to be owing to its high SiO_2 concentration, which binds the free $Ca(OH)_2$ in the mixture and induces the development of extra binder gels [18].

ASTM C 270 [22] provides information about the minimum standards that cement mortars should have, and there are four categories of standards, particularly "M," "S," "N," and "O" for various field uses and mortar kinds. The "M" group has the highest strength requirement among the classes. Mortars in this group are required to have an average compressive strength of at least 17.2 MPa in 28 days. The other "S," "N," and "O" groups have minimum average compressive strengths of 12.4 MPa, 5.2 MPa, and 2.4 MPa, respectively. Given the compressive strength values of several series in Figure 4, it is seen that all series comply with ASTM C270 [22] standards, and all series are "M" group. Therefore, it is seen that the series produced within the scope of the study comply with the standards and can be applied in real life.

Ustabaş and Ömür [13] studied the impact of obsidian substitution on the heat of hydration and mechanical characteristics of cement mortar. In this study, 10%, 20%, 30%, 30%, 40%, and 50% by weight of obsidian, blast furnace slag, and fly ash were added to cement, and the hydration temperature and 2-, 7- and 28-day compressive strengths of mortars were investigated. The highest strength value was obtained from the reference sample containing 100% cement, while the order of magnitude of the strengths obtained from 40 wt% substitutions of fly ash, blast furnace slag, and obsidian were blast furnace slag, obsidian and fly ash, respectively. When the 2-, 7- and 28-day values are analyzed, it is noteworthy that there is an inverse relationship between the percentage increase in obsidian weight and compressive strength. Within the scope of our study, a similar relationship was found in 3, 7, and 14-day strengths. In this context, the values of the study are based on the literature.

Pehlivan [23] looked into the use of nano-silica in the manufacturing of calcined cement mortars by adding 10%, 20%, and 30% calcined clay and 0%, 0.5%, and 1% nano silicate to cement and examined 360-day compressive and flexural strengths. While 55.35 MPa compressive strength and 9.43 MPa flexural strength were obtained from the control specimen containing 100% cement, the series having 10% calcined clay and 1% nano-silica had the maximum compressive strength of 58.41 MPa and flexural strength of 9.75 MPa. When the strength values are analyzed in general, it is seen that there are decreases in compressive and flexural strengths when the additive ratio exceeds 10%. Aruntaş et al. [18] In their study, they substituted 10%, 20%, and 30% by weight of ground blast furnace slag and slaked lime to determine the effect of ground blast furnace slag and slaked lime on cement mortar. Compressive and flexural strengths were seen to decrease as the replacement rate soared. In this context, it is seen that our study is compatible with the literature. Dilek and Akpınar [24] investigated the effect of using waste bricks and glass as aggregates instead of normal aggregates on cement mortars. They obtained compressive strengths of 29.3 and 36.2 MPa in mortars where 100% of waste bricks and glass were used in 28 days. Although he received 30.24% and 13.81% lower compressive strength than the control sample, he received a significant result for a sustainable future. On the other hand, economy is one of the most critical criteria for determining building materials for engineering applications. When considered in terms of economy, cement has a significant share. From this perspective, the study results are promising for the future.

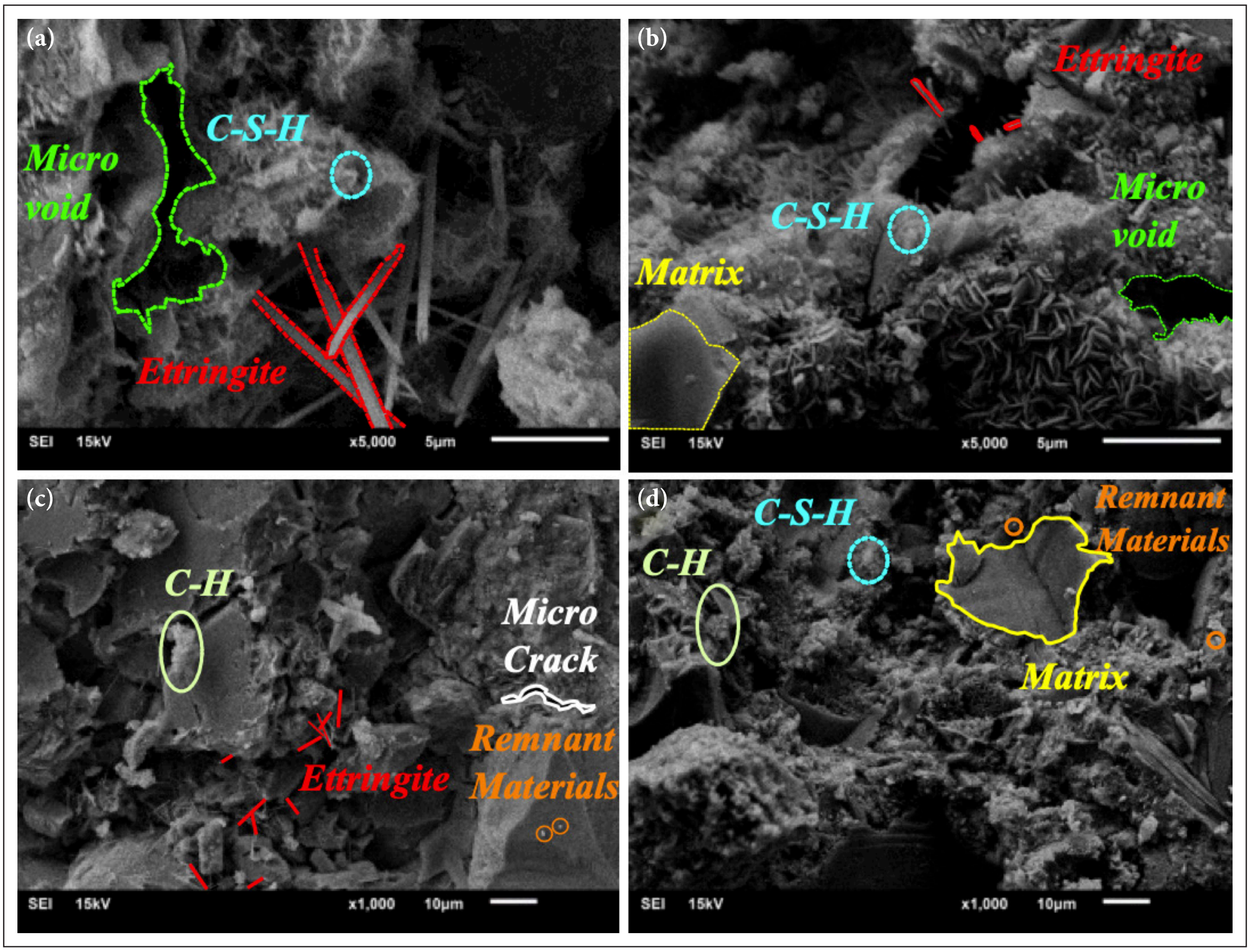

Figure 6. SEM images of S1-28 and S4-28 mortar samples.

3.2. XRD Analysis

The variables are monochromatic wavelength λ Cu-K-beta, model PW1830, 4°–90° 2Θ° angle range with automatic database for XRD analysis. 28-day-old cement and obsidian containing different mortar samples were scanned. The XRD patterns of the samples and their crystal structures are shown in Figure 5. When the model is examined in general, it is noteworthy that the most essential and central peak is Quarz (SiO_2), as well as the presence of structures such as CH-Portlandite and ettringite, which are hydration products. In the XRD model, it is seen that there are no non-hydrated materials, such as C_3S and C_2S, which are the primary base materials of the cement [25]. This indicates that the cement reacted completely and formed hydration products. Mortars containing obsidian, on the other hand, do not include any non-hydrated products. This suggests that obsidian substitution has no adverse effect on gel formation. Also, obsidian forms C-S-H gels by connecting free $Ca(OH)_2$ with its high SiO_2 content. Compared to the reference sample S1-28, the S4-28 sample with 30% obsidian substitution shows a decline in CH density and a rise in C-S-H content [26, 27]. It is thought that the primary source of quartz peaks in both mixtures in the XRD model is due to the CEN standard sand used in the mix.

3.3. SEM Analysis

28-day-old S1 mortar specimens containing 100% cement and S4 mortar specimens containing 30% obsidian substitution were analyzed using SEM to search the microstructural features. Figure 6 shows x1000 and x5000 zoomed SEM images of different regions. When the S1 sample containing 100% cement in Figure 6a is examined, the presence of microvoids, C-S-H gel, and ettringite structures in the structure is noteworthy. In Figure 6b, the structure of specimen S4 with 30% obsidian substitution shows C-S-H gel, micro-void, matrix, and ettringite structures similar to specimen S1. Etrengite is formed as a result of reactions between gypsum and aluminate phase. When gypsum is depleted, the remaining aluminate phase reacts with etrengite to form monosulfates or AFm [28]. As the density of these structures increases, the materials' mechanical and durability properties are adversely affected. When the dimensions of the structures are examined in detail, the density of ettringite in sample S4 is very low compared to S1. In this case, the compressive strengths obtained from the specimens and SEM images support each other. It is seen that the volume of microvoids in sample S4 is less than in sample S1. It is seen that the density of C-S-H gels in sample S4 is higher compared to sample S1. Figure 5c and Figure 5d show C-H (Portlandite), microcracks, matrix, ettringite, and unreacted materials in samples S1 and S4. It can be seen that the density of unreacted materials in specimen S1 is higher than S4. This is known to have a negative effect on compressive strength. Similarly, the presence of C-H in S1 is higher than in S4. Binder materials with pozzolanic properties contribute to forming new C-S-H by binding C-H [29–31]. The low C-H density in the S4 series shows a relationship between the pozzolanic properties of obsidian. Depending on this situation, the strength properties also increase. The results obtained from SEM images support the results obtained from mechanical tests.

4. CONCLUSION

This work compared the mechanical and microstructural characteristics of cement and obsidian powder substitution mortar specimens. The results obtained from the mechanical testing and microstructural analysis (SEM, XRD) of the mortar specimens helped greatly to know the link between mechanical strength and microstructural characteristics.

The SEM images of the reference specimen and obsidian-substituted mortars clearly show the linear relationship between compressive strength and parameters such as C-S-H gel density, compact structure, low ettringite density, microcracks size, etc. Furthermore, due to the high SiO_2 content of obsidian, a pozzolanic material, the high density of C-S-H peaks in sample S4 in the XRD models of mortar samples demonstrates the favorable influence of pozzolanic materials on the mechanical strength of mortars. The study results show that substituting obsidian in concrete can positively improve mechanical strength in applications where early strength is not required. It is thought that different substitution rates and durability properties of obsidian can be studied in future studies.

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ETHICS

There are no ethical issues with the publication of this manuscript.

DATA AVAILABILITY STATEMENT

The authors confirm that the data that supports the findings of this study are available within the article. Raw data that support the finding of this study are available from the corresponding author, upon reasonable request.

CONFLICT OF INTEREST

The authors declare that they have no conflict of interest.

FINANCIAL DISCLOSURE

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USE OF AI FOR WRITING ASSISTANCE

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