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Impact of Natural and Calcined Zeolite on Mortar Performance: Optimization of Cement Substitution Ratios and Calcination Temperatures

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Abstract

The environmental and economic efficacy of natural and calcined zeolite surpasses that of cement. However, due to the inherent lack of binder properties in natural and calcined zeolite, this study explores their potential as partial replacements for cement at varying ratios: 5%, 10%, 15%, and 20%. Also micronized zeolite is subjected to calcination at temperatures of 600 ℃, 700 ℃, and 800 ℃ for an hour. Consistency is maintained through the incorporation of a superplasticizer, with water/binder ratios held constant. Mortars, incorporating natural and calcined micronized zeolites, are produced in dimensions of 50x50x50 mm. The compressive strength is assessed at 7 and 28 days, complemented by microstructure analyses utilizing Scanning Electron Microscopy (SEM) and X-ray Diffraction (XRD) experiments. The experimental results indicate that the inclusion of natural and calcined zeolite with cement yields superior outcomes compared to the control mortar. Furthermore, samples containing 15% zeolite calcined at 700°C showed a notable increase in compressive strength, with approximately 12% higher strength at 7 days and 20% at 28 days compared to the control sample.

1 Introduction

The increase in world population brings forth fundamental necessities such as water, food, living spaces, and energy, posing significant challenges alongside these growing demands. Meeting the rising demand for energy has led to an increasing preference for fossil fuels over renewable energy sources, playing a crucial role in the rise of carbon dioxide (CO_2) emissions in the atmosphere. Across the globe, various measures, such as the Kyoto Protocol and similar environmental regulations, have been implemented to reduce CO_2 emissions. These efforts significantly impact the construction sector, where considerations of human health preservation, cost-effectiveness, and the efficient utilization of natural resources make it imperative to explore alternative materials.

One alternative among these resources is pozzolans. The substitution of natural or artificial pozzolans, in specific proportions with cement, holds the potential for both cost reduction and a decrease in CO_2 emissions. Pozzolans react with $Ca(OH)_2$ generated during the hydration process, forming additional binding components. This reaction enhances the impermeability of concrete and improves its resistance in chemical environments, along with improving its mechanical properties. Such characteristics play a pivotal role in the adoption of sustainable practices within the construction sector (Rodriguez et al. 2002; Papadakis and Tsimas 2002) The pozzolanic materials contained in blended cements typically consist of silica and alümina (Lea 1970; Poon et al. 1999). In this context, it is noteworthy that natural zeolites also contain substantial amounts of SiO_2 and Al_2O_3 (Canpolat et al. 2004).

Natural zeolites find applications in various industries such as pollution control, energy efficiency, mining, agriculture, and livestock due to their physical structures and chemical properties. Among many types, clinoptilolite is highly desired, alongside chabazite, analcime, erionite, ferrierite, heulandite, laumontite, mordenite, and phillipsite (Kurudirek et al. 2010). Clinoptilolite, possessing high specific surface area, exhibits increased pozzolanic reactivity (Ghafari et al. 2016). The utilization of zeolite as a mineral additive is attributed to its notable features, encompassing a high ion exchange capacity, abundant silica content, low density, elevated specific surface area, and a crystal structure conducive to dehydration. These features

contribute to zeolite being an outstanding material (Sevim and Okumuş 2011). Additionally, the exceptional porosity and reduced density inherent in natural zeolites render them suitable for application as lightweight construction materials within the construction industry. Zeolite, being a softer material compared to Portland cement clinker, facilitates the grinding process more easily and quickly due to its porous structure (Canpolat et al. 2004). However, the naturally occurring voids in zeolite lead to an increased water demand in the mixture. Therefore, the use of a superplasticizer chemical additive is necessary to maintain consistent cement paste (Fragoulis et al. 1997; Quanlin and Naiqian 2005). These characteristics make zeolite an environmentally friendly and energy-efficient alternative in various industrial applications.

Natural zeolites are crystalline aluminosilicate minerals that share similar chemical compositions with clay minerals but exhibit a three-dimensional structure in contrast to the layered structure of clay minerals. Natural zeolites are known to possess significant pozzolanic activity. Due to this property, various researchers have investigated the potential use of natural zeolites as a sustainable construction material in blended cements and concrete mixtures (Snellings et al. 2009; Perraki et al. 2010; Janotka et al. 2003; Perraki et al. 2003; Kontori et al. 2009; Martínezet al. 2006; Ceylan et al. 2021; Kitsopoulos and Dunham 1996; Kop and Yazıcıoglu 2024)

Zeolite, with abundant global reserves and high pozzolanic activity, has been utilized as a mineral additive in cement and concrete production facilities across several countries, including Russia, Germany, Slovenia, Cuba, Serbia, Spain, and China in recent years (Nas and Kurbetci 2018). Kucukyildirim and Uzal (2014) investigated the use of zeolite calcined at 200°C, 400°C, and 600°C for 2 hours in high-performance pozzolan-modified cements. They observed that calcined zeolites exhibited lower water demand and improved compressive strength performance compared to raw zeolite. Additionally, they noted that the cementitious system with reduced porosity demonstrated higher compressive strength.

Burris and Juenger (2020) examined changes in phase content, particle size, surface area, and pore size distribution of various natural clinoptilolite zeolites through calcination at temperatures ranging from 300°C to 965°C. Their study indicated that calcination increased the particle size and amorphous content of zeolite while reducing its porosity and surface area. These findings offer key observations for understanding the potential and performance of natural and calcined zeolite in cement and concrete applications.

In a study conducted by Dayı et al. (2013) the physical and mechanical properties of composite cements produced by incorporating various proportions of zeolite, fly ash, waste glass, and Portland cement clinker were examined, and the potential use of these materials in composite cement production was investigated. Overall, the experimental results indicated that the physical and chemical properties of the produced cements met the relevant standards, and it was determined that zeolite at a 10% replacement ratio could be used in the production of Portland composite cement.

In this study, unlike previous research, micron-sized ground zeolite is utilized, and a broader range of cement replacement ratios (5%, 10%, 15%, and 20%) along with various calcination temperatures (600°C, 700°C, and 800°C) are investigated. This comprehensive approach provides new and significant insights into the use of zeolite as a cement replacement by thoroughly examining both microstructural transformations and mechanical properties. Through determination of an optimal amount of natural and calcined zeolite in

replacement with the cement, this study aims to: 1) improve mechanical and microstructural properties of mortars, 2) develop a more environmentally friendly mortar by reducing cement content in production, and 3) lower construction costs. Conducted experiments aim to identify the optimal mixture ratio and calcination temperature for mortar samples among the different ratios used in the experimental series. The elevated SiO_2 content in zeolite plays a crucial role in the hydration process. The interaction between the $Ca(OH)_2$ compound present in this mineral and its transformation into C-S-H gel contributes to the improvement of mechanical properties of mortar.

2 Material and Method

2.1 Material

Cement: Ordinary Portland Cement (OPC) CEM I 42.5R conforming to the TS EN 197-1 (TS EN 197-1 2012) standard is utilized for all specimens. A comprehensive overview of the chemical and physical properties of the cement sample is provided in Table 1. The initial and final setting times of the cement are determined as 165 and 280 minutes, respectively.

Zeolite: The zeolite employed in the experiments is of the clinoptilolite type, sourced from Gördes, Manisa. The micronized zeolite's laser particle size distribution was acquired using the Mastersizer device, as illustrated in Fig. 1. The chemical and physical properties specific to clinoptilolite zeolite are depicted in Table 1, while images of both natural and calcined zeolites are presented in Fig. 2.

As illustrated in Fig. 1, the laser particle size distribution analysis of micronized zeolite reveals that 86.25% of the material is below 10 microns.

Table 1
Physical and chemical compositions of cement and zeolite (Kop and Yazıcıoglu 2024)

Component (%)	Cement	Zeolite(Gördes)
SiO_2	19.05	70
CaO	60.91	3.44
Al_2O_3	5.31	8.64
Fe_2O_3	3.08	2.14
MgO	2.15	1.2
SO_3	3.56	1.2
Na_2O	0.87	1.14
K_2O	1.35	2.04
Specific Gravity	3.11	2.3
Specific Surface Area (cm^2/g)	3640	19340

Plasticizer: To counteract potential adverse impacts on consistency that may arise from zeolite addition and to ensure workability, a superplasticizer is incorporated into mortar mixtures at varying rates, ranging from 0.8–1.5% of the binder weight. In this context, the chemical properties of the sodium naphthalene sulfonate-based liquid superplasticizer are detailed in Table 2.

Table 2
Superplasticizer Properties

Solid Content Ratio	pH (2% solution)	Sodium Sulfate Content	Viscosity	Specific Gravity
Min. 38%	6.5–9	0,50%	Max 150 cP (25°C)	1.20 gr/cm³ ± 0.05

Sand: In all mortar samples, CEN standard sand has been used as a fine aggregate TS EN 197-1 (TS EN 197-1 2012).

2.2 Method

The calcination process is conducted in an ash furnace with a temperature capacity of 1200°C and a heating rate of 20°C/min. Zeolites prepared for calcination are placed in ceramic containers and heated for 1 hour. Upon completion of the heating period, the samples are removed from the furnace and spread onto large metal trays to facilitate rapid cooling in air. Mortar samples are subjected to cement substitution with natural and calcined micronized zeolite at rates of 5%, 10%, 15%, and 20%. Furthermore, micronized zeolite underwent calcination at temperatures of 600 ℃, 700 ℃, and 800 ℃ for one hour and is employed as a

substitute for cement. Mortar production utilized CEN standard sand, and its particle distribution is outlined in Table 3.

Table 3
CEN standard sand grain distribution

Square Mesh Size (mm)	Cumulative Retained on Sieve (%)
2	0
1.6	7 ± 5
1.00	33 ± 5
0.50	67 ± 5
0.16	87 ± 5
0.08	99 ± 5

The binder/aggregate ratio in mortar mixtures is consistently maintained at 1/3, and the water/binder ratio is held at 0.50. Mortars incorporating natural and calcined zeolite substitutions exhibited reduced workability. Efforts are made to sustain workability by incorporating a superplasticizer. A total of 17 experimental groups are prepared for the study, and the mixture ratios are detailed in Table 4.

Table 4
Mortar Mixture Ratios

Serial	Portland Cement (g)	Zeolite (g)	Calcined Zeolite (g)			Sand (g)	Water / Binder	Water (g)	Superplasticizer (g)
			600℃	700℃	800℃				
Z0	450	0	0	0	0	1350	0.5	225	0
Z5	427.5	22.5	0	0	0	1350	0.5	225	3.6
Z10	405	45	0	0	0	1350	0.5	225	4.65
Z15	382.5	67.5	0	0	0	1350	0.5	225	5.7
Z20	360	90	0	0	0	1350	0.5	225	6.75
C6Z5	427.5	0	22.5	0	0	1350	0.5	225	3.6
C7Z5	427.5	0	0	22.5	0	1350	0.5	225	3.6
C8Z5	427.5	0	0	0	22.5	1350	0.5	225	3.6
C6Z10	405	0	45	0	0	1350	0.5	225	4.65
C7Z10	405	0	0	45	0	1350	0.5	225	4.65
C8Z10	405	0	0	0	45	1350	0.5	225	4.65
C6Z15	382.5	0	67.5	0	0	1350	0.5	225	5.7
C7Z15	382.5	0	0	67.5	0	1350	0.5	225	5.7
C8Z15	382.5	0	0	0	67.5	1350	0.5	225	5.7
C6Z20	360	0	90	0	0	1350	0.5	225	6.75
C7Z20	360	0	0	90	0	1350	0.5	225	6.75
C8Z20	360	0	0	0	90	1350	0.5	225	6.75

The workability values of the prepared mortar mixtures are consistently maintained with a fixed spread diameter of 165 ± 10 mm, as per TS EN 1015-3 (TS EN 1015-3 2000), utilizing a flow table apparatus. Fresh mortars are cast into cubic molds with dimensions of 50x50x50 mm. These cast samples are demolded after 24 hours and subjected to water curing at 20 ± 2°C until the testing days. Subsequent to the 7 and 28 days curing processes, the compressive strength of five 50x50x50 mm mortar samples for each test series is measured in accordance with TS EN 196-1 (TS EN 196-1 2016), as depicted in Fig. 3. Additionally, microstructural properties are analyzed through X-ray Diffraction (XRD) and Scanning Electron Microscopy (SEM) analyses.

3 Results and Discussion

3.1 Workability of Fresh Cement Mortar

In this study, the workability of cement mortars incorporating various proportions of zeolite and calcined zeolite as replacements is assessed by measuring the spread values of fresh mortars using the flow table test. The flow table test results for all mixtures are presented in Fig. 4.

The results of the flow table test indicated that increasing the zeolite content had an adverse effect on the workability of the mortar. To mitigate this negative impact, a superplasticizer additive is incorporated. The sample images of the flow table test are presented in Fig. 5.

3.2 Compressive Strength

Compressive strength tests at 7 and 28 days are conducted on 50x50x50 mm samples incorporating natural and calcined zeolite substitutions for cement. Each test series comprised five samples. While samples substituted with natural zeolite exhibited lower early strengths compared to the control sample, they demonstrated higher strengths in later stages. Specifically, natural zeolite substituted mortars with serial numbers Z5, Z10, and Z20 displayed lower compressive strengths than the control sample at 7 days. The Z15 mixture exhibited results approximately similar to the control sample at 7 days, with a compressive strength value of 42.52 MPa. Subsequently, natural zeolite substituted mixtures (Z5, Z10, Z15, Z20) achieved higher compressive strengths at 28 days compared to the control sample. Among these mixtures, Z15 mixture demonstrated the highest strength, recording a value of 57.02 MPa.

Mortars incorporating calcined zeolite substitutions demonstrated superior compressive strength values compared to those with natural zeolite substitutions. Notably, among the calcined zeolite substituted mortars, the C7Z15 mixture exhibited the most favorable results. The 7 day compressive strength of this mixture measured 47.26 MPa, and the 28 day compressive strength measured 61.06 MPa. Comprehensive compressive strength values for the samples at both 7 and 28 days are illustrated in Figs. 6 and 7.

In the study conducted by Florez et al. (2021), it was reported that the fineness of grinding enhances compressive strength. The 28-day compressive strength results obtained for mortar mixtures incorporating raw zeolite in this study align with the findings of Ramezanianpour et al. (2013)d çükyıldırım and Uzal (2014).

3.3 Scanning Electron Microscope analysis

The acquisition of images in the Scanning Electron Microscope (SEM) entails the focalization of a high-voltage accelerated electron beam onto the sample, followed by the scanning of this beam across the sample surface. During this process, the effects generated by the interaction between the electrons and the sample atoms are collected by appropriate detectors. Subsequently, this accumulated information is transmitted to the screen of a cathode ray tube, having passed through signal amplifiers.

To characterize the microstructure of samples with a 15% substitution of natural and calcined zeolite, as well as control samples, SEM analysis is conducted. Figures 8 and 9 present SEM analyses of mortars with natural and calcined zeolite substitutions. The results of the examination reveal the presence of calcium silicate hydrate (C-S-H) and calcium hydroxide (portlandite), which are primary hydration products of cement, in the analyzed samples. Notably, more pores and voids are observed in the control mortars. Moreover, an increase in C-S-H gels is observed in mortars with calcined zeolite substitution. Hence, it is presumed that

mortars with calcined zeolite substitution exhibit improved mechanical properties as a result of these observed changes. Similarly, Li et al. (2023) observed a reduction in voids in the microstructure and an increase in the formation of C-S-H gels with zeolite substitution.

The reaction of zeolite with calcium hydroxide ($Ca(OH)_2$) leads to the formation of C-S-H (calcium silicate hydrate) gels with siliceous or siliceous and aluminous materials that possess hydraulic binding properties. High SiO_2 content can support the formation of C_3S and C_2S compounds, thereby enhancing pozzolanic properties and contributing to the formation of additional calcium silicate hydrate (C-S-H) gels (Kop and Yazıcıoglu 2024; Demir et al. 2020,2018; Simsek et al. 2022; Guzelkucuk et al. 2020)

3.4 X-ray Diffraction analysis

XRD operates by irradiating a sample with X-rays and analyzing the resulting diffraction patterns caused by the scattering of X-rays off the atoms within the material. The angles and intensities of these diffracted X-rays provide valuable insights into the interatomic distances within the crystal lattice and the elemental composition of the sample. Consequently, this data enables the identification of crystalline phases, determination of crystal structures, and quantification of phase proportions within the sample by diffracting scattered X-rays at specific angles, which are then recorded by a detector. Analysis results obtained through the powder X-ray diffraction technique are compared with peaks identified using prepared catalogs to determine the presence of specific minerals. In addition to microscopic analyses, XRD analysis provides a suitable method for the mineralogical characterization of different types of aggregates (Gullu and Kadioglu 2017).

X-ray diffraction patterns of clinoptilolite zeolite samples following natural and calcination processes are presented in Fig. 10. Natural clinoptilolite zeolite exhibits crystalline properties, characterized by sharp peaks. The XRD analysis indicates that the mineral composition of natural zeolite comprises approximately 75–80% clinoptilolite, 10–15% quartz and 5–10% feldspar. The X-ray diffraction analysis of the samples, as denoted by the characteristic narrow and intense peaks at $2\theta = 9.84°$, 22.44°, 26.06°, and 30.07°, illustrates the well-crystallized nature of clinoptilolite by displaying the positions of corresponding characteristic peak points (Korkuna et al. 2006; Petrov et al. 1995; Nezamzadeh-Ejhieh and Moeinirad 2011).

With an increase in the degree of calcination, a gradual decrease in the intensities of clinoptilolite peaks has been observed. Post-calcination processes, the XRD pattern still exhibits the presence of clinoptilolite peaks; however, their intensities have significantly diminished, indicating a substantial collapse of the crystal lattice. Comparable changes in clinoptilolite following thermal treatment have been reported by other researchers as well (Christidis et al. 2003; Duvarcı et al. 2007; Dimowa et al. 2013; Akkoca et al. 2013; Alver et al. 2019)

4 Conclusions

In this study, compressive strength and microstructural properties of mortar samples incorporating natural and calcined zeolite as a cement substitute are systematically explored. Following results are obtained:

1. The specimens containing 15% zeolite, calcined at 700 ℃, demonstrate superior performance in both early and late strength when compared to the control sample and natural zeolite. A notable

enhancement of approximately 12% in compressive strength at 7 days and around 20% at 28 days is observed in comparison to the control sample.
2. Mortars incorporating 15% natural zeolite exhibit comparable results to the control sample at 7 days, but a significant increase of approximately 12% in compressive strength is observed at 28 days compared to the control sample.
3. XRD analyses reveal that the calcination process lead to a reduction in the density of clinoptilolite peaks, disrupt the crystal structure, and transform it into an amorphous structure.
4. The calcination process conducted at 700°C significantly enhances the pozzolanic activity, consequently improving the mechanical and physical properties of mortar. However, at a higher calcination temperature, 800°C, a diminishing effect on pozzolanic activity is observed.
5. Using zeolite instead of cement in mortar results in a reduction in specific gravity with increasing zeolite content, due to the lower specific gravity of zeolite compared to cement. Conversely, as the specific surface area of zeolite is higher than that of cement, replacing cement with zeolite results in an amplification in specific area with increasing zeolite content.
6. The substantial SiO_2 content in zeolite facilitates the conversion of the calcium hydroxide (CH) compound into the C-S-H gel during the hydration process, contributing significantly to the improvement of mechanical properties in the mortar.

As a result, the transformation of zeolite into an amorphous structure through calcination, its grinding to micron size, and its high SiO_2 content have enhanced its pozzolanic activity, thereby improving the strength and microstructural properties of mortar samples.

Declarations

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Author Contribution

M.K. Literature review, conducting experiments, analyzing results, and paper composition.S.Y. Literature review, analyzing results, and paper composition.

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Figures

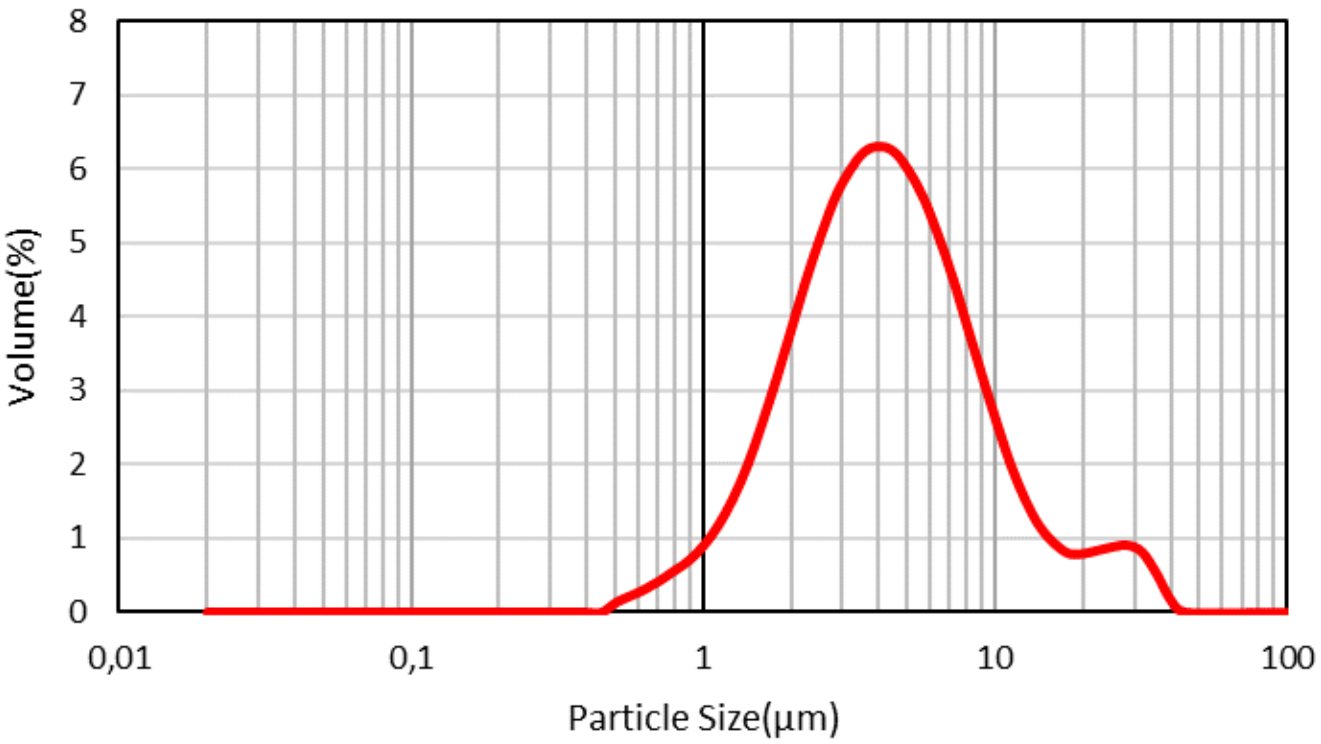


Figure 1

Zeolite laser particle size distribution (Kop and Yazıcıoglu 2024)



Figure 2

Natural and calcined zeolite

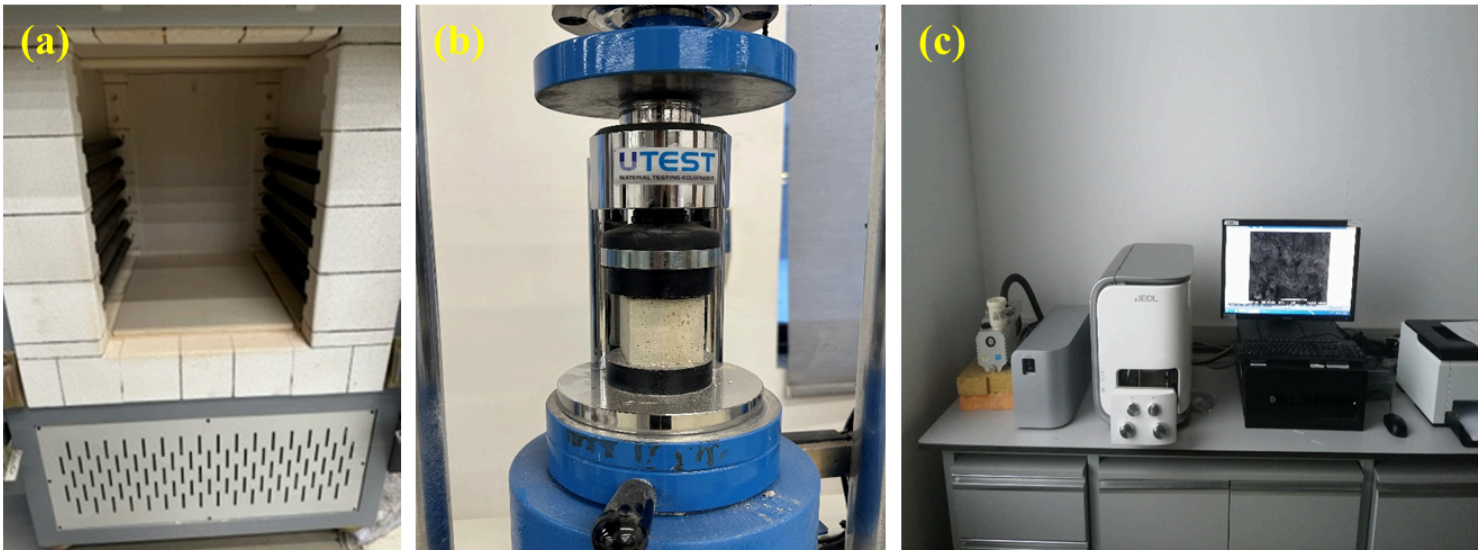


Figure 3

(a) Ash Furnace, (b) Compressive Strength Testing Machine, (c) JEOL Scanning Electron Microscope

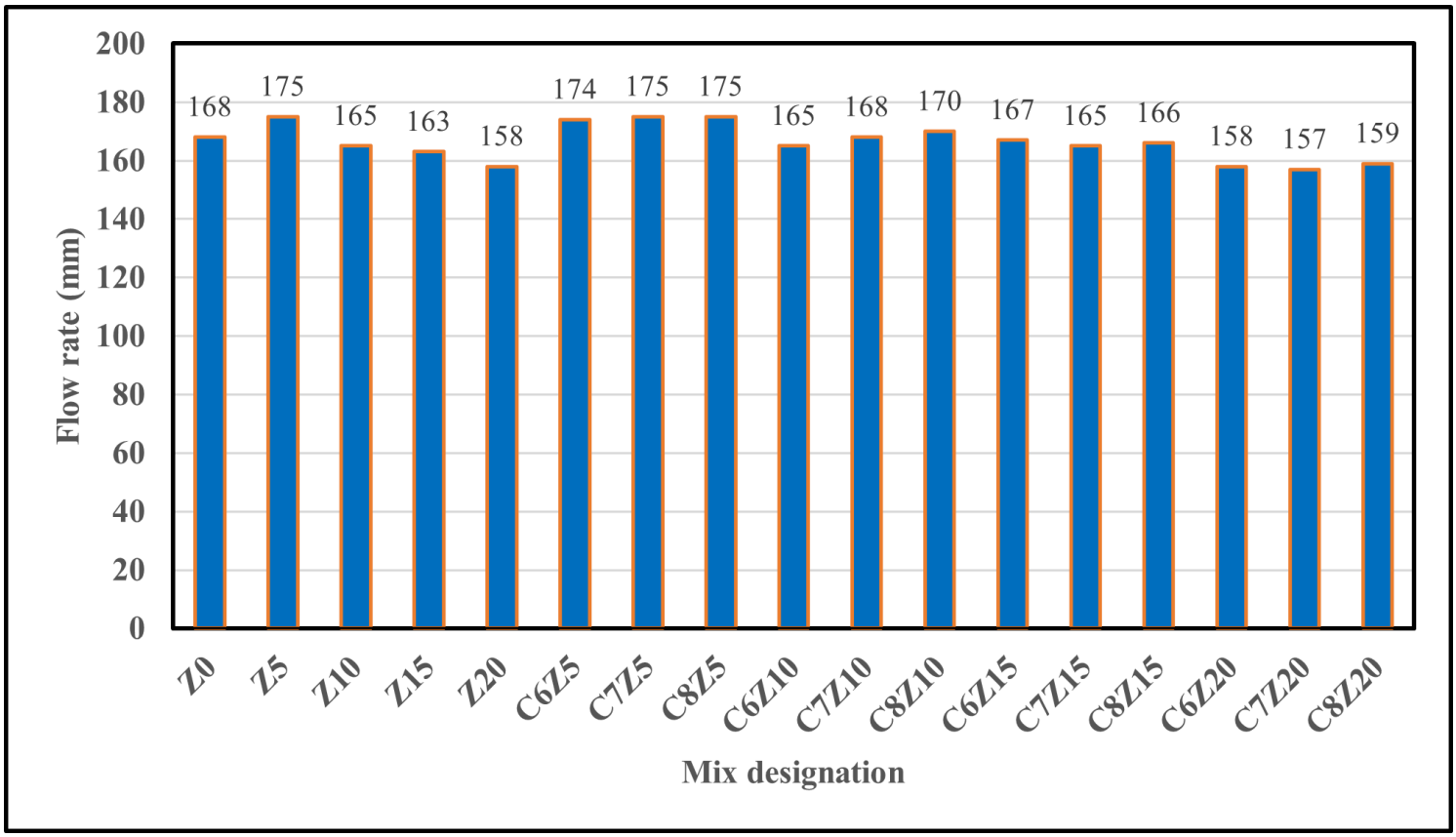


Figure 4

Flow rate results of fresh mortars.



Figure 5

Mortar flow method

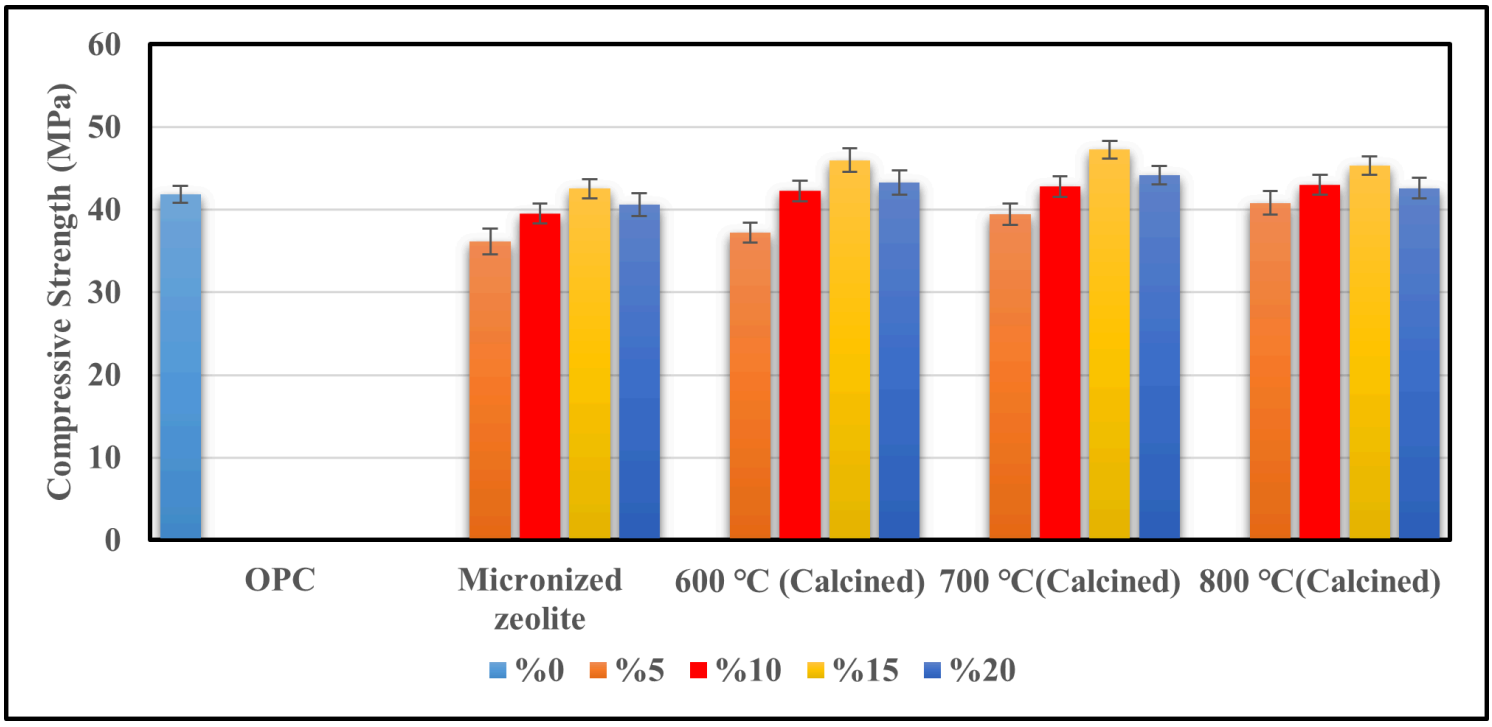


Figure 6

7 Day Compressive Strength

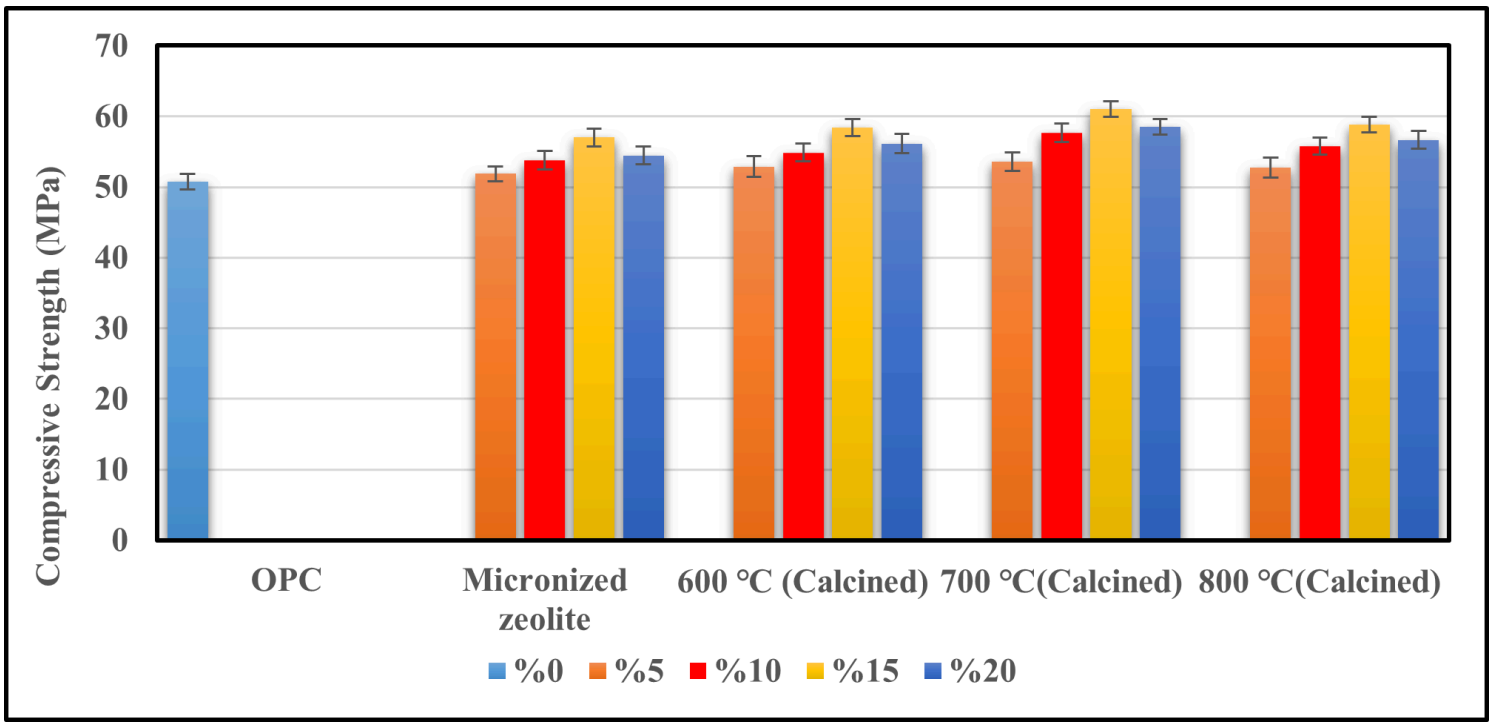


Figure 7

28 Day Compressive Strength

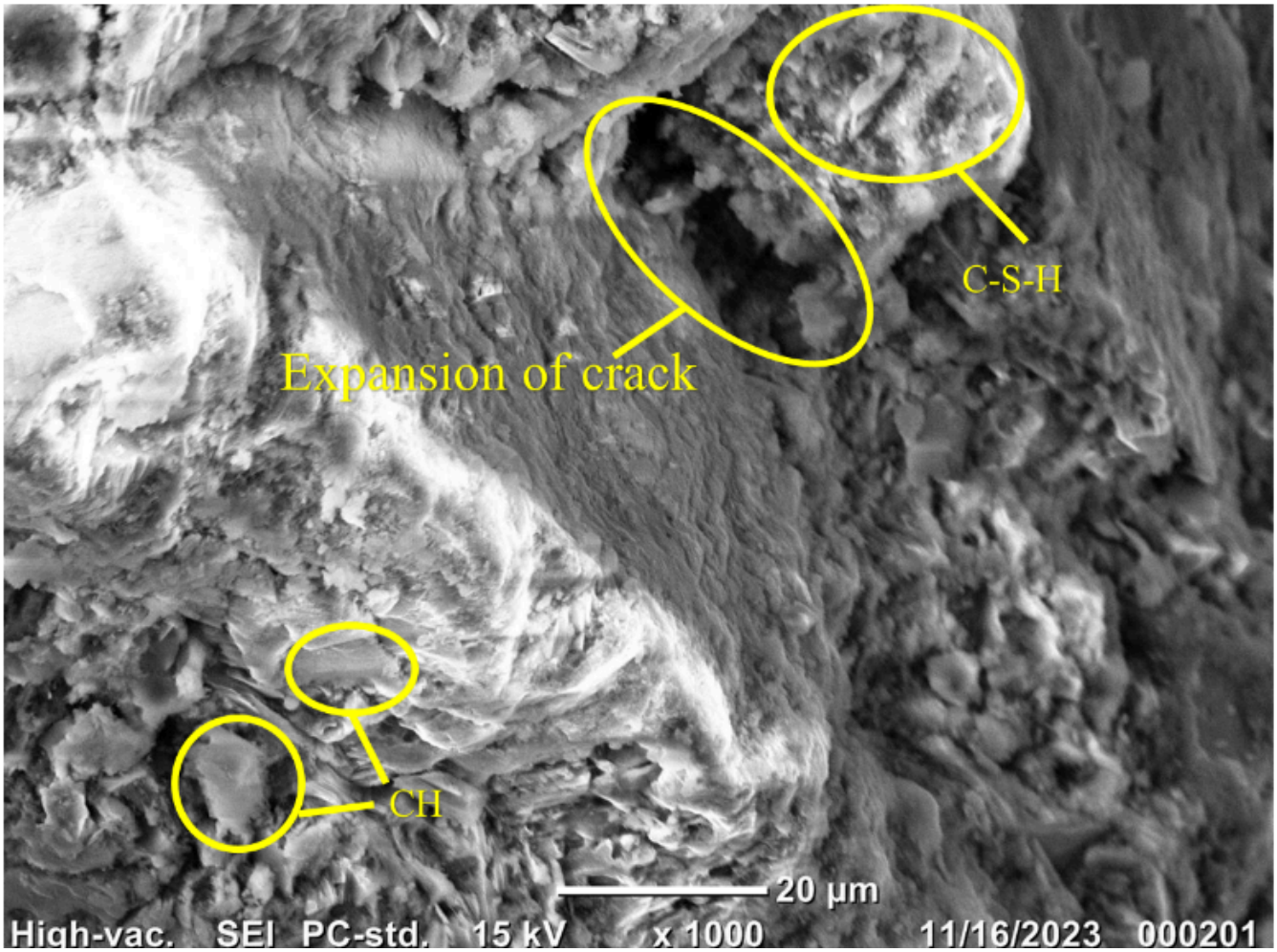


Figure 8

SEM Analysis of Control Sample

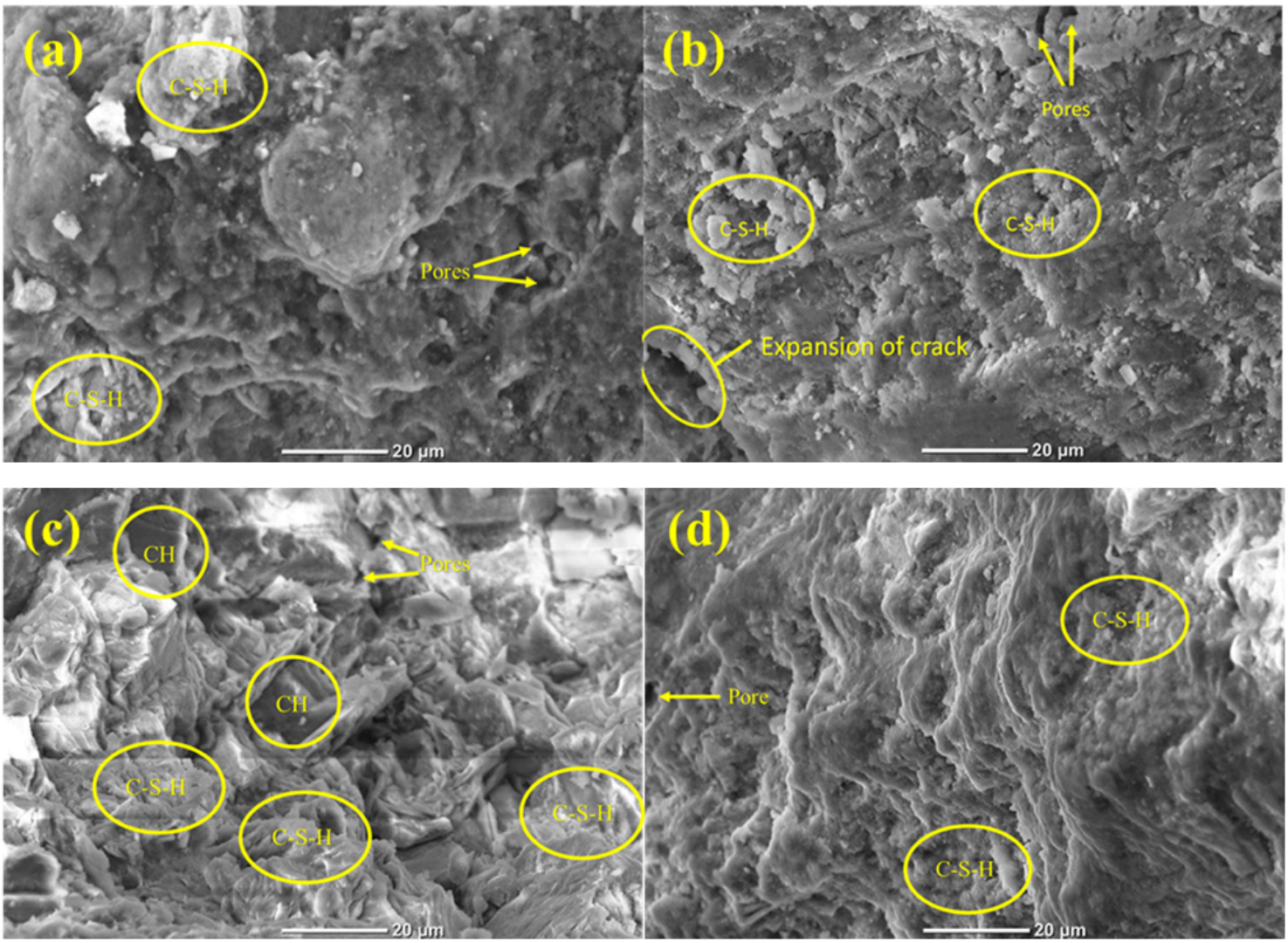


Figure 9

SEM Analysis (a) Z15, (b) C6Z15, (c) C7Z15, (d) C8Z15