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Research Article

INVESTIGATION OF THE EFFECT OF CALCINATION TEMPERATURE AND FINENESS OF KAOLIN FROM BALIKESİR REGION ON POZZOLANIC ACTIVITY

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Keywords

Kaolin,
Metakaolin,
Fineness,
Compressive Strength,
Pozzolanic Activity.

Abstract

The use of mineral additives in cement production supports an environmentally friendly production process by reducing the amount of clinker and significantly reducing CO_2 emissions. Methacholine, obtained as a result of calcination of kaolin at high temperatures, is used effectively as a pozzolan. The study was carried out in two stages to determine the effect of calcination temperature and fineness on the pozzolanic activity index. In the first stage, kaolin was calcined at 3 different temperatures: 600°C, 750°C and 900°C. Mortar samples in accordance with the standard were prepared to determine the pozzolanic activity strength index depending on the calcination temperature. When the 28-day pozzolanic activity strength index results were examined, it was seen that kaolin calcined at 600°C provided the best strength value of 122%. In the second part of the study, the material calcined at 600 °C was ground to three different finenesses as 13800 cm^2/g, 15900 cm^2/g and 18700 cm^2/g, and mortar samples were prepared to determine the pozzolanic activity strength index. As the fineness increased, the pozzolanic activity strength index values also increased.

BALIKESİR BÖLGESİNE AİT KAOLİNİN KALSİNASYON SICAKLIĞININ VE İNCELİĞİNİN PUZOLANİK AKTİVİTEYE ETKİSİNİN İNCELENMESİ

Anahtar Kelimeler

Kaolin,
Metakaolin,
İncelik,
Basınç Dayanımı,
Puzolanik Aktivite.

Özet

Mineral katkıların çimento üretiminde kullanılması klinker miktarını azaltırken CO_2 emisyonlarını önemli ölçüde azaltarak çevre dostu bir üretim sürecini desteklemektedir. Kaolinin yüksek sıcaklıklarda kalsinasyonu sonucu elde edilen metakolin, puzolan olarak etkili bir şekilde kullanılmaktadır. Çalışma kalsinasyon sıcaklığının ve inceliğin puzolanik aktivite indeksine etkisini belirlemek amacıyla 2 aşamada gerçekleştirilmiştir. İlk aşamada kaolin 600°C, 750°C ve 900°C olmak üzere 3 farklı sıcaklıkta kalsine edilmiştir. Kalsinasyon sıcaklığına bağlı olarak puzolanik aktivite dayanım indeksini belirlemek için standarda uygun harç numuneleri hazırlanmıştır. 28 günlük puzolanik aktivite dayanım indeksi sonuçları incelendiğinde %122 ile en iyi dayanım değerini 600°Cde kalsine edilen kaolinin sağladığı görülmüştür. Çalışmanın ikinci kısmında 600 C° de kalsine edilen malzeme 13800 cm^2/g, 15900 cm^2/g ve 18700 cm^2/g olmak üzere 3 farklı incelikte öğütülmüş ve puzolanik aktivite dayanım indeksinin tespiti için harç numuneleri hazırlanmıştır. İnceliğin artmasıyla birlikte puzolanik aktivite dayanım indeksi değerlerin de artış görülmüştür.

Alıntı / Cite

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Highlights

- Investigation of the effects of calcination of kaolin at different temperatures
- Determination of the role of fineness on pozzolanic activity
- Investigation of the use of kaolin material in concrete in Balıkesir region.

Graphical Abstract

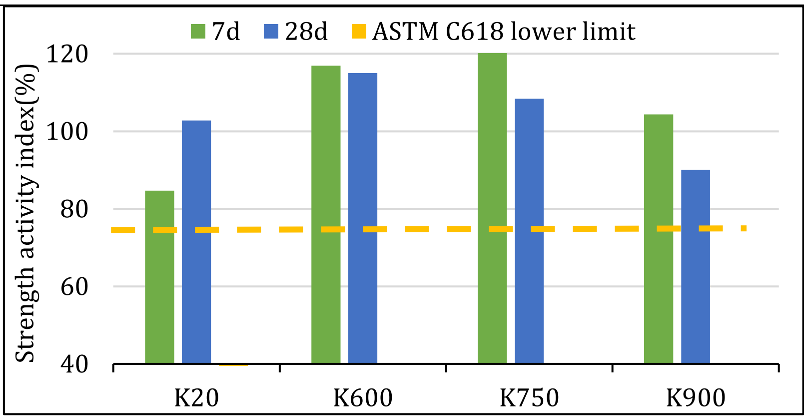

Figure. 7 and 28 Strength activity index values

Purpose and Scope
This study aims to determine the effect of kaolin calcined at different temperatures and fineness on the pozzolanic activity index.

Design/methodology/approach
The effect of kaolin calcined at different temperatures and fineness on the pozzolanic activity index was determined by testing the compressive strength.

Findings
According to the results of compressive strength tests, it was determined that kaolin calcined at different temperatures and fineness affected the pozzolanic activity index.

Research limitations/implications
In future studies, it is recommended to investigate the effect on the pozzolanic activity index by using different calcination methods.

Social Implications
It is predicted that the use of kaolin as a mineral additive can reduce CO_2 emissions in clinker production.

Originality
This study presents a more effective usage method by revealing the effect of kaolin calcined at different fineness and temperature on the pozzolanic activity index.

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1. Introduction

Nowadays, increasing environmental awareness and sustainability targets lead to significant changes in the construction and building materials industry (Yılmaz and Bakış, 2015). In this context, cement production draws attention as one of the largest sources of carbon dioxide emissions worldwide. The high amount of energy consumption and CO_2 emission in the cement production process necessitates the development of more environmentally friendly and sustainable production methods in the sector (He et al., 2019).

In cement production, pozzolans are widely used additives to improve environmental sustainability and product performance (Tulashie et al., 2021). Pozzolans are silica or alumina-containing materials that do not exhibit binding properties on their own but gain binding properties with cement when they react with water. They are divided into 3 main categories as natural pozzolans (such as volcanic tuffs and diatomites), artificial pozzolans (such as fly ash, blast furnace slag and silica fume) and processed natural pozzolans (such as calcined clay) (Erdoğan, 2007). Pozzolans provide chemical and physical advantages in the cement matrix. Chemically, pozzolans react with calcium hydroxide released during hydration reactions to form more durable calcium silicate hydrate (C-S-H) phases, which increases the long-term durability of cement (Şişman et al., 2022). Physically, the fine-grained structure of pozzolans helps to improve the microstructure of concrete and reduce the void ratio. This increases impermeability and durability. Furthermore, the use of pozzolans provides a significant environmental benefit in cement production. While a large amount of CO_2 is emitted in the production of traditional Portland cement, pozzolans offer an environmentally friendly alternative with a low carbon footprint (Ceylan, 2020).

Clays undergo significant chemical and physical changes at high temperatures, making them valuable material for various industrial applications (Astudillo et al., 2023). When heated at high temperatures (generally in the range of 600-900°C), the bound water molecules in the internal structure of clays are released and the mineral structure of the clay is transformed (Hollanders et al., 2016; Gültekin and Ramyar, 2022). In this process, the aluminum silicate minerals in the clay content turn into an amorphous structure, which makes the clay reactive. This transformation at high temperature increases the usability of clays as pozzolans in cement production (Snellings et al., 2023). When calcined clay is included in the cement matrix, it reacts with calcium hydroxide released during the hydration process to form more durable calcium silicate hydrate (C-S-H) phases (Özcan and Güngör, 2019). Calcined clay obtained by heating clays at high temperatures offers significant advantages when used as a binding material in concrete and mortar mixtures. In addition, the calcination process increases the surface area and reactivity of the clay, helping to reduce the amount of energy used in cement production and CO_2 emissions (Erdemoğlu et al., 2019; Karatas et al., 2020). High temperature calcined clays play an important role not only in the cement industry but also in other industries such as ceramics, refractory materials and catalyst carriers. The mechanical and thermal durability of the clay increases with the effect of high temperatures, which makes calcined clays stand out in applications requiring high performance. As a result, calcination of clays at high temperatures is of great importance for both sustainable material production and industrial efficiency.

In this study, the effect of thermal treatment on the strength activity index of kaolin obtained from the Balıkesir region was investigated. Kaolin samples were calcined at 600°C, 750°C, and 900°C in a furnace in accordance with ASTM C311 standards, and the optimal calcination temperature was determined based on the findings. Additionally, the calcined kaolin was ground to three different fineness levels of 13800 cm^2/g, 15900 cm^2/g, and 18700 cm^2/g using a ball mill, and the effect of fineness on the strength activity index was thoroughly examined.
The results indicate that kaolin from the Balıkesir region can be utilized as metakaolin, and its use as a partial replacement for cement has the potential to enhance strength properties while contributing to a reduction in CO_2 emissions. This study provides significant insights into the sustainable utilization of local resources and offers valuable data for the effective use of Balıkesir kaolin by determining its optimal calcination temperature.

2. Material and Methods

In the study, kaolin clay was crushed in a jaw crusher, dried to constant mass at 105°C, ground in a ball mill and calcined at 3 different temperatures (600°C, 750°C and 900°C) for 1 hour. The material was then allowed to cool to room temperature within 24 hours. In order to determine the effect of calcination on the pozzolanic activity index, the compressive strength of mortar samples produced with non-calcined (K20) and calcined kaolin additives was determined on days 7 and 28. According to the results obtained, the optimum calcination temperature was determined. The material was then used in 13800 cm^2/g, 15900 cm^2/g and 18700 cm^2/g finenesses. These fines were milled for 3 different times (120, 150 and 180 min.) and pozzolanic activity indices were determined.

After the grinding and calcination processes of kaolin material, the specific surface area was determined by Blaine device at Süleyman Demirel University (SDU) Natural and Industrial Building Materials Application and Research Centre (DEYMAM) in accordance with TS EN 196-6 standard. Specific surface area measurement is shown in Figure 1. Specific surface area was determined using Equation 1 and Equation 2.

$$S = \frac{K}{\rho} \times \frac{\sqrt{e^3}}{(1-e)} \times \frac{\sqrt{t}}{\sqrt{10-\eta_1}} \quad (1)$$

$$S = \frac{524.2K \times \sqrt{t}}{\rho} \quad (2)$$

S, Blaine fineness cm^2/g; K, device constant; η, viscosity of the air at the experimental temperature (20 ±2 ˚C) (Pa.s); e, porosity of the bed; t: time, s; ρ: density of the powder material g/cm^3 ; for the value e=0.500; Equation 2 was used.

Figure 1. Blaine test

CEM I 42.5 R type cement obtained from Isparta Göltaş Cement Company was used in the preparation of mortar specimens. The chemical content of CEM I 42.5 R type cement is given in Table 1 (Göltaş, 2024).

Table 1. Chemical and physical properties of cement

Chemical Component	Content (%)	Physical and Mechanical Properties	
SiO_2	19.30	Fineness (45 μ)	0.50
Al_2O_3	3.95	Fineness (90 μ)	3.50
Fe_2O_3	3.40	Blaine (cm^2/g)	4100
CaO	63.0	Specific Gravity (g/cm^3)	3.14
MgO	1.80	Le Chatelier (mm)	0,60
SO_3	3.00	Setting Time Initial (dk)	170
CI	0.15	Setting Time Final (dk)	265
Free CaO	1.50	2 Days (MPa)	24.5
LOI	3.80	28 Days (MPa)	49.5

In the preparation of the mortars, tap water from the city of Isparta was used. For the production of the samples, TS EN 196-1 CEN standard sand with a density of 2.56 g/cm^3 was used. The granulometry of the standard sand is given in Table 2.

Table 2. Granulometry of standard sand (TS EN 196-1)

Sieve Aperture (mm)	Cumulative Remaining in Sieve (%)
2.00	0.00
1.60	7 ± 5
1.00	33 ± 5
0.50	67 ± 5
0.16	87 ± 5
0.08	99 ± 1

The prepared mortar mixture was placed in 5×5×5 mm mortar molds according to ASTM C311. For each series, 3 samples were cured in lime-saturated water at 20±1°C until the test date. Before measurement, the samples were removed from the water and left to dry to be ready for testing. According to the findings obtained from the tests, the 7-day and 28-day compressive strength of the samples were calculated using Equation 3 according to the TS EN 196-1 standard.

$$f_c = \frac{P}{A} \qquad (3)$$

f_c, compressive strength, N/mm^2; P, axial compressive force, N; A, area of the sample, mm^2.
The pozzolanic activity strength indices were determined using Equation 4 according to ASTM C311 (ASTM, 2022).

$$PAI = \frac{A}{B} \times 100 \qquad (4)$$

PAI, pozzolanic activity strength index, %; A, average compressive strength of the test sample, N/mm^2; B, average compressive strength of the control sample, N/mm^2.

The prepared mortar samples and compressive strength tests are shown in Figure 2.

Figure 2. Fresh mortar specimens (left) and compressive strength test (right)

3. Experimental Results

The kaolin used in the study was obtained from a company operating in the Balıkesir-Sındırgı region. The chemical composition of the kaolin was determined using the XRF method at Afyon Kocatepe University (AKÜ) Accredited Natural Stone Analysis Laboratory (DAL), and the findings are presented in Table 3.

Table 3. Chemical composition of kaolin

Chemical Component	Content %
SiO_2	46.75
Al_2O_3	35.53
Fe_2O_3	0.835
CaO	0.46
MgO	0.03
K_2O	0.56
Na_2O	1.23

The specific gravity of the kaolin used in the production of the mortar samples was determined using the Micromeritics Accupyc II 1340 gas pycnometer device located at SDÜ DEYMAM. In the measurement, ASTM D 5550-06 standard was referenced, a 10 cm^3 measuring cup was used, and for each non-calcined and calcined sample, 5 repetitions were performed, with the average values presented in Table 4. Additionally, the Blaine test findings are presented in Tables 5 and 6.

Table 4. Average mass values of kaolin samples determined by gas pycnometer

Kaolin Clay	Density (g/cm^3)
K20	2,64
K600	2,68
K750	2,69
K900	2,71

Table 5. Blaine values of kaolin grinded at constant time and calcined at different temperatures

K	ρ	η_1	e	t	S
22.492	2.64	0.00001819	0.495	1015	13747
22.492	2.64	0.00001819	0.488	1045	13491
22.492	2.64	0.00001819	0.497	1015	13886
Average (K20)					**13708**
22.492	2.68	0.00001819	0.490	1025	13403
22.492	2.68	0.00001819	0.504	1000	14197
22.492	2.68	0.00001819	0.496	1020	13812
Average (K600)					**13804**
22.492	2.69	0.00001819	0.492	1035	13547
22.492	2.69	0.00001819	0.498	1010	13822
22.492	2.69	0.00001819	0.506	990	14203
Average (K750)					**13857**
22.492	2.71	0.00001819	0.493	1080	13829
22.492	2.71	0.00001819	0.497	1065	14017
22.492	2.71	0.00001819	0.494	1080	13882
Average (K900)					**13909**

Table 6. Blaine values of kaolin clays grinded at different times and calcined at 600°C

K	ρ	η_1	e	t	S
22.492	2.68	0.00001819	0.490	1025	13403
22.492	2.68	0.00001819	0.504	1000	14197
22.492	2.68	0.00001819	0.496	1020	13812
Average (B1)					**13804**
22.492	2.68	0.00001819	0.489	1480	16025
22.492	2.68	0.00001819	0.484	1535	15893
22.492	2.68	0.00001819	0.485	1504	15797
Average (B2)					**15905**
22.492	2.68	0.00001819	0.499	1860	18890
22.492	2.68	0.00001819	0.493	1890	18439
22.492	2.68	0.00001819	0.496	1900	18769
Average (B3)					**18700**

The water requirement of the mortar mixtures was determined using a Vicat needle according to ASTM C618 Standard. Accordingly, the quantities of materials used in the production of the mortar mixtures are given in Table 7.

Table 7. Components of reference and kaolin added mortars

Specimens	Cement (g)	Kaolin (g)	Sand (g)	Water (g)
Reference	500	-	1375	242
K20	400	100	1375	244
K600	400	100	1375	245
K750	400	100	1375	246
K900	400	100	1375	247
B1	400	100	1375	245
B2	400	100	1375	246
B3	400	100	1375	247

K* Calcination temperatures. Numbers are degrees of temperature.
B* Blaine values. 1-2-3 symbolizes increasing fineness.

3.1 The Effect of Calcination Temperature on Mortar Compressive Strength

The results of compressive strength tests applied to samples obtained at 7 and 28 days within the scope of the study are presented in Figure 3. When examining the effects of using calcined kaolin mineral in concrete on strength, it was observed to increase strength compared to the reference sample. Upon examining the maximum strengths at 7 and 28 days, the highest compressive strength at 7 days was achieved at 750°C, measuring 46.08 MPa, while at 28 days, the highest compressive strength was recorded at 600°C, measuring 54.03 MPa. Consequently, based on the evaluation using 28-day compressive strength values, a calcination temperature of 600°C is found to be suitable.

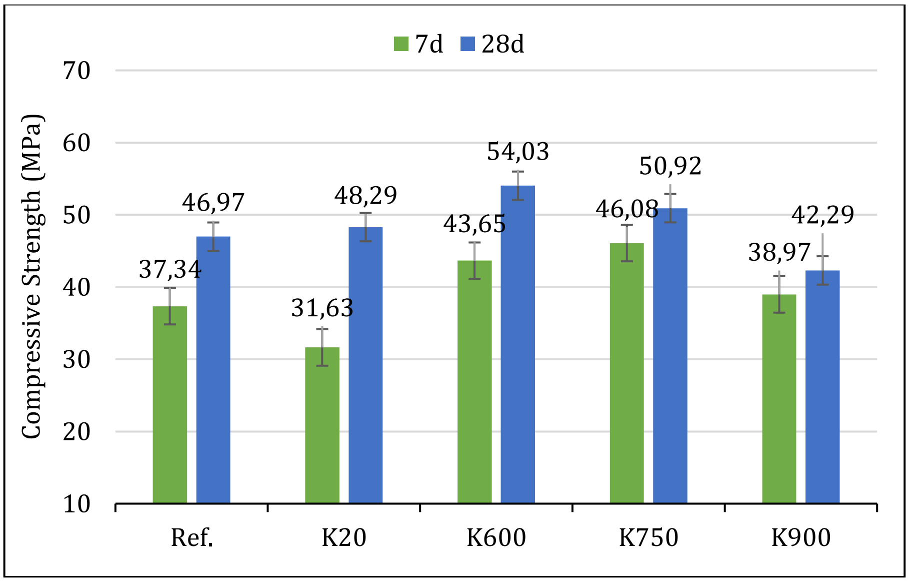

Figure 3. Compressive strength values of mortar specimens at 7 and 28 days

According to ASTM C618 standard, the minimum PAI (pozzolanic activity index) for pozzolanic materials is 75% at 7 and 28 days. In this study, kaolin samples calcined at different temperatures exhibit PAI values above 75% (Figure 4).

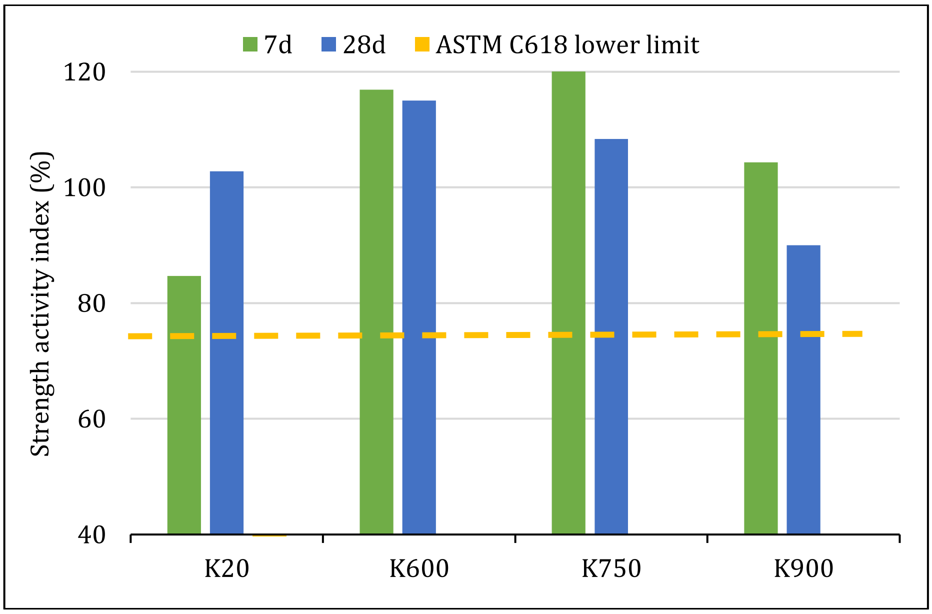

Figure 4. Temperature dependent variation of 7 and 28 days pozzolanic activity index values

3.2 The Effect of Specific Surface Area on Pozzolanic Activity

In the study, based on the findings, the most suitable calcination temperature was determined to be 600°C. The kaolin clay subjected to calcination at this temperature was ground at different durations in a ball mill, and their specific surface areas were determined using the Blaine test device according to TS EN 196-6 standard. The specific surface areas of the samples were calculated as 13800 cm^2/g (B1), 15900 cm^2/g (B2), and 18700 cm^2/g (B3). The effects of these three differently ground calcined kaolin samples on compressive strength and PAI values are presented in Figures 5 and 6.

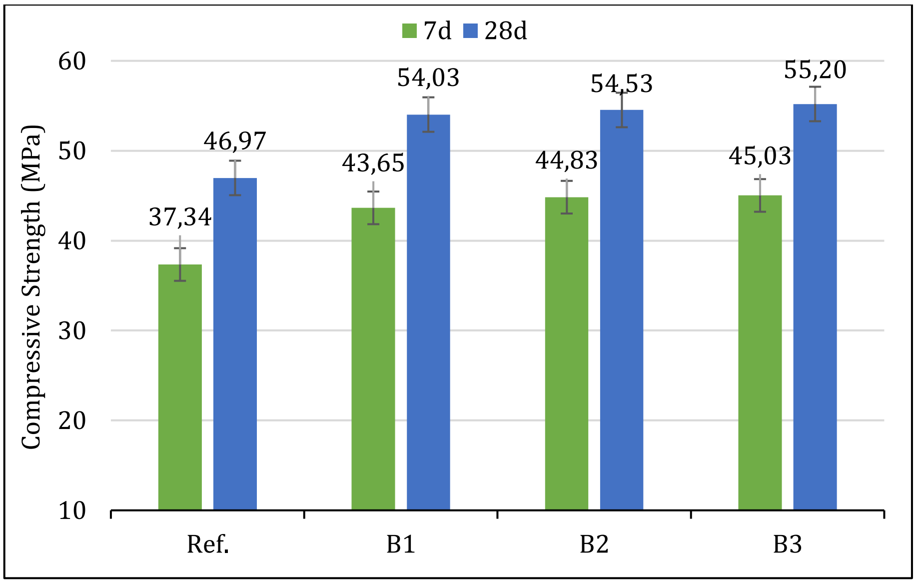

Figure 5. Compressive strength values of mortar specimens at 7 and 28 days

According to the experimental results, all mortar samples exhibited higher compressive strength compared to the reference sample. The highest compressive strength, 55.20 MPa, was achieved in sample B3. However, the strength values are very close to each other. When evaluated in terms of strength activity index, it was determined that calcined kaolin samples with three different fineness levels meet the requirements of ASTM C618 standard.

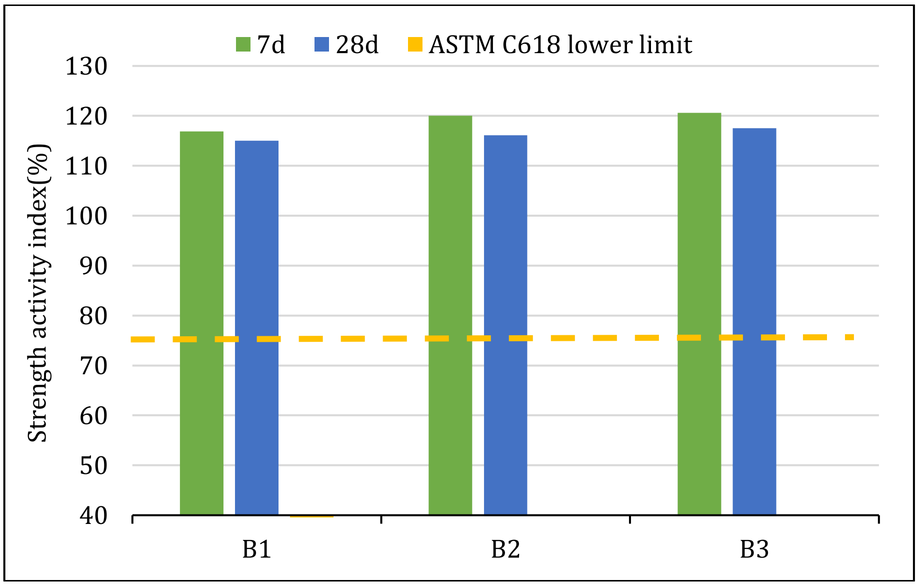

Figure 6. Variation of 7 and 28 day pozzolanic activity index values depending on different finenesses

4. Discussion and Conclusion

In this study, variations in the pozzolanic activity index of kaolin clay according to ASTM C618 standard were investigated as a function of calcination temperature and fineness. The changes observed in kaolin after calcination at different temperatures, along with the effects of material fineness levels on compressive strength and pozzolanic activity, were evaluated. The results indicate that calcination temperature has a more significant effect on the pozzolanic properties and mechanical performance of kaolin. Detailed analysis of the results revealed the following:

- No significant change was observed in specific surface areas of kaolin ground to a constant fineness before and after heat treatment.
- The optimal calcination temperature for mortar compressive strength was determined to be 600°C.
- Mortars produced with kaolins of different fineness at the optimal calcination temperature resulted in a 13% and 35% increase in pozzolanic activity index, corresponding to a 1% and 2.2% increase in compressive strength, respectively.
- Due to the pozzolanic reactivity of calcined clay, the use of kaolin as an additive improved the compressive strength of mortars by 14% and 25% at 7 and 28 days, respectively.

The test results suggest that despite the increase in specific surface area, the lack of significant increase in compressive strength indicates that maximum pozzolanic reactivity has been achieved (Siline, M., & Mehsas, B., 2022). Similar conclusions were drawn in this study as well. Compressive strength results at specific surface areas of 13800, 15900, and 18700 cm^2/g were consistently close at 7 and 28 days. Therefore, economically speaking, calcined kaolin at approximately 14000 cm^2/g fineness and 600°C provides a sufficient pozzolanic activity index ratio. The utilization of kaolin from the Balıkesir region as a partial replacement for cement in varying proportions and the investigation of its activity factor would be a valuable area of study.

Conflict of Interest

The authors have not declared any conflict of interest.

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