Investigation of the Pozzolanic Properties of Basic Mining Slages Exposed to Heat Treatment

Isıl İşleme Maruz Kalmış Bazik Maden Cüruflarının Puzolanik Özelliklerinin Araştırılması

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ÖZET

Dünya üzerinde artan nüfus ve beraberinde getirdiği inşaat ve çimento kullanım ihtiyacı gün geçtikçe artmaktadır. Çimento üretimi aşamalarında doğa verilen zarar ve harcanan enerjinin önlenmesi için çimento yerine kullanılabilecek ürünler araştırılmaktadır. Çimento yerine kullanılabilen malzemelere puzolan denmektedir, puzolanlar doğada var olan ya da sonradan aktifleştirilen ince taneli yapılardır. Doğal yollarla elde edilmeyen puzolanların büyük bir çoğunluğu cüruf şeklinde bulunmaktadır. Metalürjik işlemler sonrası oluşan maden atıklarına ergitme cürufu denir. Isıl ve geri kazanım işlemleri sonucunda elde edilen bu ürünler atık olmakla birlikte ikincil işlemler ile ekonomiye kazandırılabilecek ikincil bir ham madde kaynağıdır. Günümüzde sıklıkla puzolan olarak kullanılan en büyük kaynaklar yüksek fırın cürufları ve çelik cüruflarıdır.

Bu çalışmada, eski maden cüruflarının ısıl işlem geçirmiş atığının puzolanik aktivitesinin belirlenmesi amaçlanmıştır. Maden atık sahasından temin edilen numuneler çimento boyutuna öğütülmüşlerdir. Amasya yöresinde bulunan maden atığının puzolanik özellikleri ve öğütüle bilirliğinin incelenmesi amacı ile farklı devir ve sürelerde (20, 30, 40, 60, 90 ve 120 dk.) öğütülerek farklı boyutlara indirilmiştir. Elde edilen ürünler elek analizi yapılarak kimyasal içerik, süre ve devir ilişkisi ortaya konmuştur. Hangi sürede ve devirde tanecik boyutunun düşeceği ve puzolanik aktivitenin artacağı önemlidir. Daha sonra puzolanik aktivitelerinin belirlenebilmesi amacıyla farklı oranlarda çimentoya puzolan ikameli karışımları hazırlanarak 7/28 günlük kür süreleri sonunda dayanım aktivite indeksleri belirlenmiştir. Çalışma sonucunda 700 dv./dak. öğütme işleminde bilye sayısının öğütme işlemini etkilemediği, 1000 dv./dk. öğütme hızının maden cürufunu önemli oranda kırılmasına ve özgül yüzey alanı değerlerinde önemli yükselişler görülmüştür.

Anahtar Kelimeler: Cüruf, dayanım aktivite indisi, maden atıkları, puzolanik aktivite, mekanik özellik

ABSTRACT

The increasing population in the world and the need for construction and cement use are increasing day by day. Products that can be used instead of cement are being researched in order to prevent the damage to nature and the energy spent during the cement production stages. Materials that can be used instead of cement are called pozzolans, they are fine-grained structures that exist in nature or are activated later. Most of the pozzolans that are not obtained naturally are in the form of slag. Mine wastes formed after metallurgical processes are called smelting slag. These products, which are obtained as a result of thermal and recovery processes, are waste, but they are a secondary raw material source that can be brought into the economy by secondary processes. The major sources frequently used as pozzolans today are blast furnace slag and steel slag. In this study, it was aimed to determine the pozzolanic activity of heat-treated waste from old mine slags. Samples obtained from the mine waste site were ground to cement size. In order to examine the pozzolanic properties and grindability of the mine waste in the Amasya region, it was reduced to different sizes by grinding at different cycles and times (20, 30, 40, 60, 90 and 120 minutes). The obtained products were analyzed by sieve analysis and the relationship between chemical content, time and cycle was revealed. It is important in which time and cycle the particle size will decrease and the pozzolanic activity will increase. Then, in order to determine the pozzolanic activities, mixtures with pozzolan substitutes were prepared for cement in different ratios, and strength activity indexes were determined at the end of 7/28 day curing periods. As a result of the study, 700 dv./min. in the grinding process, the number of balls does not affect the grinding process, 1000 dv./min. It was observed that the grinding speed significantly reduced the slag and the specific surface area values increased significantly.

Keywords : Slag, strength activity index, mine waste, pozzolanic activity, mechanical property

1. INTRODUCTION

Cement is the most costly of the materials used in concrete production. Cement production also causes serious environmental problems due to intense energy consumption and CO_2 emissions. For these reasons, innovative and sustainable studies are needed. In these studies, the use of pozzolans is in the first place. Pozzolan has little or no self-binding properties but they are siliceous or siliceous and aluminous materials that can gain binding property by chemically reacting with calcium hydroxide if they are ground very finely and in a humid environment. Pozzolans taken directly from nature are called natural, and those that are activated by chemical treatment are called artificial pozzolans. Natural pozzolans are those that are found in nature and are used either by pre-treatment or by grinding directly. Volcanic ash, volcanic tuffs, tras etc. can be given as examples of this group. Artificial pozzolans are by-products of energy sources used in the processing of raw materials in industry. Fly ash, blast furnace slag, silica fume and the like can be given as examples of artificial pozzolans (Kulekci and Cullu, 2019). Pozzolanic activity is defined as the ability of active silica in pozzolan to react with $Ca(OH)_2$ and water. As a result of this reaction, in addition to the C-S-H gels formed by the hydration of the cement, new C-S-H gels are formed despite the use of much less cement and an increase in strength occurs (Erdogan, 2013). In other words, at the end of this reaction, while the amount of unreacted portlandite ($Ca(OH)_2$) decreases, free CHs react with pozzolans and increase the amount of calcium silica hydrate (C-S-H) (Kulekci et al., 2021; Massazza, 1993). In order to determine the pozzolanic activity, chemical and mechanical test methods are included in the standards ASTM C311 (2005) and TS 25 (2008). In the chemical test method, silica and $Ca(OH)_2$ are determined qualitatively and quantitatively. In the mechanical test method, the flexural and compressive strengths of the mortars produced with pozzolans mixed with lime or cement are determined and it is investigated whether the pozzolans have active silica content (Kulekci, 2021c; Cullu et al., 2013; Kılınckale, 1996). Pozzolans form calcium silicate hydrates by entering the following chemical reaction in hydrated medium between the reactive silica they contain and the free lime formed as a result of cement hydration.

$$2Ca(OH)_2 + SiO_2 + H_2O \rightarrow 2CaOxSiO_3xnH_2O \quad (1)$$

This reaction is known as the basic pozzolanic reaction. As a result of this reaction, which develops very slowly, hydrated products similar to the silicate components of portland cement are formed. However, this reaction takes a long time to both wait for free lime formation and to gain strength with the pozzolanic effect as a result of a very slow reaction (Yalcın and Guru, 2006). Pozzolans are expected to have a better internal structure and strength by showing a good reaction in a long time (Cullu et al., 2016). This study, which aims to provide scientific data on their use with optimum values in the cement industry in Türkiye, was primarily carried out in order to ensure the active use of local and natural resources that can contribute to sustainable cement in a pozzolanic sense. Therefore, the aim of the study is to determine the physical and chemical properties of the ancient mine slag obtained from the Amasya region and to examine whether it has the potential to be used as a pozzolan in sustainable cement production. In order to determine their pozzolanic activities, samples were prepared by substituting 0, 10, 20, 30 and 40 % pozzolan to the cement and strength activity indexes were determined at the end of the 7/28 day curing period. The data obtained, the pozzolanic activity characteristic of the slag was revealed.

2. MATERIAL AND METHOD

2.1. Material

Cement, standard sand and heat treated basic mine slag were used in the study. In addition, water that does not contain harmful organic matter and mineral salts was used in the production of all samples.

2.1.1 Cement

CEM I 42.5 R type portland cement (PC) supplied from Gümüşhane cement factory was used as cement in the experiments. The chemical compositions of CEM I 42.5 R Cement are given in Table 1 and other physical properties such as setting time, compressive strength and loss of heating are given in Table 2.

2.1.2 Standard Sand

In the pozzolanic activity tests, natural silica sand, which is called CEN standard sand, preferably round grained and with a silicon dioxide content of at least 98%, was used. The chemical compositions of standard sand are given in Table 1.

2.1.3. Heat Treated Slag

The ores of the mine slags used in our studies were obtained from the Amasya region. Six separate mine slags (Figure 1) from different parts of the old mine site have different chemical components and different grades.

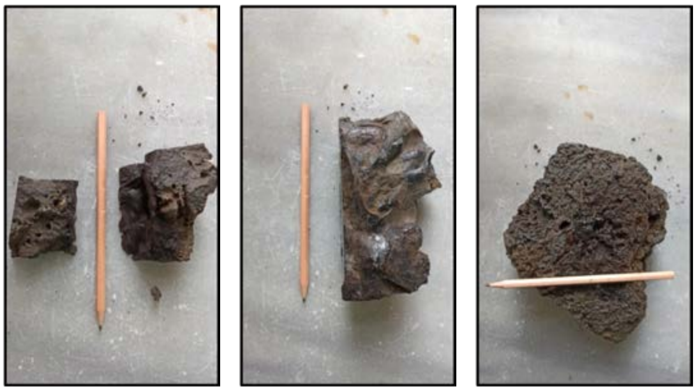

Figure 1. The pictures of samples which were taken from six seperate mine slags

Table 1. Chemical properties of raw materials.

Raw Materials	SiO_2	Al_2O_3	Fe_2O_3	MgO	CaO	Na_2O	K_2O	Total
Standard Sand	92.1	2.02	0.43	0.03	0.2	0.93	1.3	97.01
Cement	19.71	4.67	3.25	1.70	63.12	0.25	0.93	93.63

Table 2. Properties of CEM I 42,5 R Cement

Physical Properties	Limit Values	Compressive Strength (MPa)		Chemical Properties	Limit Values
Initial Setting Time (minute)	≥ 60	2 Days	≥ 20	Loss of Glow	≤ % 5,0
Expansion (mm)	≤ 10	7 Days	-	Insoluble Residue	≤ % 5,0
		28 Days	≥ 42,5	Sulfate (SO_3)	≤ % 4,0
				Chloride Content	≤ % 0,10

2.2. Method

It is known that the specific surfaces (Blaine), densities, chemical compositions and mineralogical structures of these materials have an extremely important effect on the pozzolanic activity in the use of ore slags as pozzolans (Akgun and Yazıcıoglu, 2017) The following methods were used to determine these parameters affecting the pozzolanic activity of the ore slag used in the study.

2.2.1. Chemical Analysis

Chemical analysis was carried out in Gümüşhane University (Türkiye) and ACME laboratory (Canada) in order to determine the composition of the samples taken from the study area, to determine the elements that make up the ore and their percentages.

2.2.2. Size Reduction Process

The grinding process takes place in the grinding medium as a result of the contact between the mill wall and the material. The grinding process occurs with the relative movement between the particles and the grinding medium. This movement can be characterized as fracture primarily caused by impact or fracture primarily caused by crushing and abrasion (King, 2001). In this study, mine slags from the old mine site were crushed up to -2.0 mm and +2.0 mm in a jaw crusher, then ground in a ball mill at different speeds and times. This material, with a maximum grain size of 2 mm, was milled to increase its fineness. The grinding process was carried out in a steel ball mill for 1 kg of slag in 2 hours; in accordance with the TS 25 standard, it is finely ground to leave a maximum of 10% residue on a 90 µm sieve. In the study, 700-1000 and 1250 rpm. 3 different grinding cycles and 6 different grinding times were used (Table 3).

Table 3. Grinding cycles and times of samples

Sample Name	Number of Revolution (min/rev)	Grain size fed into the mill	Grinding time (min)
C1	1000	-2mm	20
C2	1000	-2mm	30
C3	1000	-2mm	40
C4	700	+2mm	60
C5	700	+2mm	90
C6	1250	+2mm	120

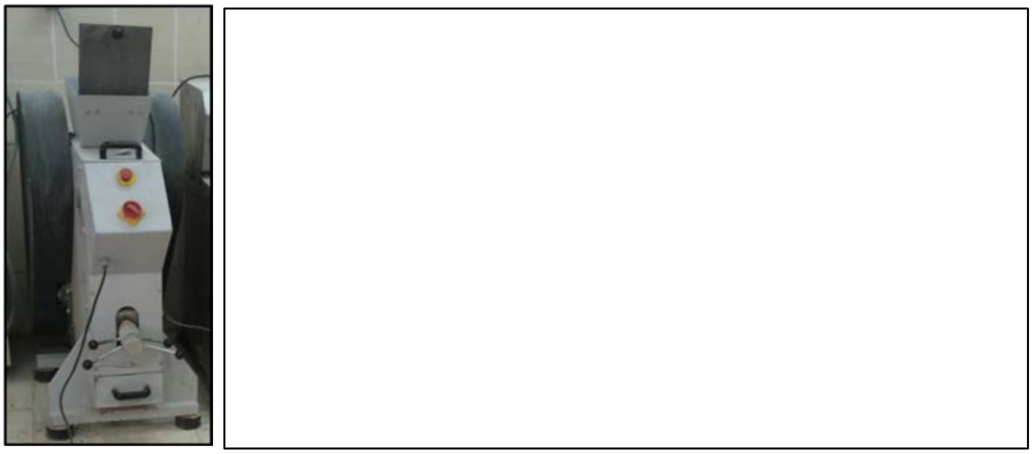

Figure 2. Crushing and grinding process of mineral slag

2.2.3. Dimensional Analysis

Particle size analysis is carried out to determine the dimensional properties of particles and is one of the frequently used measurements in many engineering and industrial fields such as ceramics, sand, clay, cement, powder metallurgy, food, pharmacology, cosmetics, paint, sedimentology and soil mechanics (Top, 2010). In this study, wet sieving method was used (Figure 3). Sieve top values were determined by Alpine brand LS-200 model device using 32, 45 and 90 µm sieves. The sample is placed on the top sieve and the sieve set is subjected to vacuum application for 10 minutes. When using small-diameter sieves for particle size analysis, a 10 g powder sample is usually sufficient. After completion of the vacuum process, the amount of powder in each size range is weighed and the percentage in the range is calculated for each portion. At this stage; the powder passing through a sieve is coded as (-) sign, and the remaining on the sieve is coded as (+) sign.

Figure 3. Sieve device used in dimensional analysis

2.2.4. Sample Production

For the pozzolanic activity test, mortars were prepared by taking different ratios of slag (0%, 10%, 20%, 30% and 40%). For each mortar group, as specified in the TS 25 Tras standard, 3 square prism samples of 40x40x160 mm dimensions were prepared (Figure 4), and the test program was created by accepting the arithmetic average of these 3 samples for the test results, and it is indicated in Table 4.

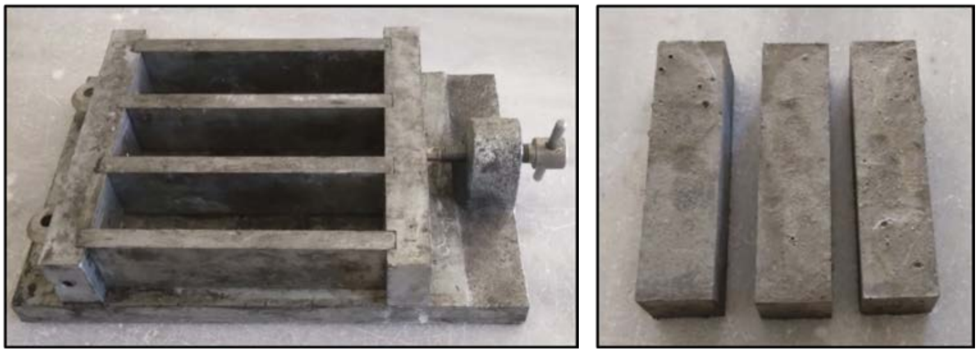

Figure 4. Prism Samples and Molds used in Flexural and Compressive Strength tests

Table 4. Experiment Schedule

Experiment aim	Experiment type	Heat (°C)	Time (day)	Number of Samples
Pozzolanic activity experiment	Mortar mix defined in TS 25	21	7	-
			28	-
Investigation of the effect of mortar component ratios on pozzolanic activity	%0 Slag by weight	21	7	3
			28	3
	%10 Slag by weight		7	3
			28	3
	%20 Slag by weight		7	3
			28	3
	%30 Slag by weight		7	3
			28	3
	%40 Slag by weight		7	3
			28	3
Total				30

2.2.5. Compressive Strength Measurement

Compressive strength tests were carried out using the MFL System brand test device, according to TS EN 12390-3 and TS EN 196-1, using broken pieces in the flexural strength test. The loading speed of the device was taken as 0.05 kN/s and the same loading speed was used in all experiments.

2.2.6. Bending Strength Measurement

Flexural strength tests were applied to 7/28 days old samples. The bending pressure press was adjusted in accordance with TS-3114 and breaking loads and compressive stresses (bending and compressive strengths) were determined by breaking automatically. The pozzolanic activity is determined as a result of experiments with the pozzolanic material.

The pozzolanic activity of the slag at different rates was determined by conducting experiments on mixtures prepared with reference pozzolan at 0, 10, 20, 30 and 40 mass percent according to ASTM C 618 standard and only on mixtures prepared with reference cement. Pozzolanic activity is expressed by calculating a value called the "resistance activity index".

Strength activity index = (A/B) x 100

Here;

A: Average Compressive Strength of Mortar Samples with Pozzolan,

B: Average Compressive Strength of Control Mortar Samples

3. RESULTS AND DISCUSSION

3.1. Chemical Composition of Heat Treated Basic Mineral Slag

The chemical compositions of the mineral slags (C1, C2, C3, C4, C5 and C6) used in the ball mill grinding tests are given in Table 5.

Table 5. Chemical compositions of the slag used in the experiments

Sample Name	SiO_2 (%)	Al_2O_3 (%)	Fe_2O_3 (%)	CaO (%)	MgO (%)	SO_3 (%)	K_2O (%)	Na_2O (%)
C1	43,52	7,87	19,91	25,32	0,92	0,11	0,59	0,26
C2	39,13	9,82	26,81	19,36	0,75	0,18	1,20	0,26
C3	47,92	7,15	17,43	23,99	0,75	0,12	0,74	0,42
C4	37,61	9,46	25,28	20,15	0,76	0,14	1,20	0,27
C5	47,93	7,19	17,13	24,36	0,74	0,13	0,74	0,41
C6	43,96	7,98	19,92	24,90	0,89	0,10	0,57	0,29

In the samples taken from the ancient mine site, it was observed that the average iron content was around 20%, the highest iron content was in the C2 sample with 26.81%, and the least content was in the C5 sample with 17.13%.

3.2. Dimensional Analysis

When we examine the surfaces of the process elements in macro dimension (Figure 5), it is seen that the mill and ball surfaces are quite clean. If an effective fracture could be created under 700 rpm cycle conditions, the ball and grinding bowl surfaces would have been plastered by the crushed mineral slag powders. An important result obtained in the grinding 700 rpm cycle conditions is that when the grinding speed is too low to activate the crushing process, the increase in grinding time and ball powder weight ratio has a very low effect on the crushing of slag powders. The particle size values corresponding to the change in mechanical grinding parameters are given in Table 6.

Figure 5. Grinding Bowl and Grinding Ball Surfaces

Table 6: Process parameters and particle size variation.

Sample No	Grinding Time(min.)	Grinding Speed(rpm)	Sieve Size (µm)	
			32	**90**
C1	20	1000	64,8	30,8
C2	30	1000	51,4	28,1
C3	40	1000	57,2	23,5
C4	60	700	53,5	22,6
C5	90	700	64,3	38,0
C6	120	1250	58,1	32,8

As a result of the sieve analysis performed after the grinding process was completed, the effect of grinding time and chemical content of the basic mine wastes on the size change was observed.

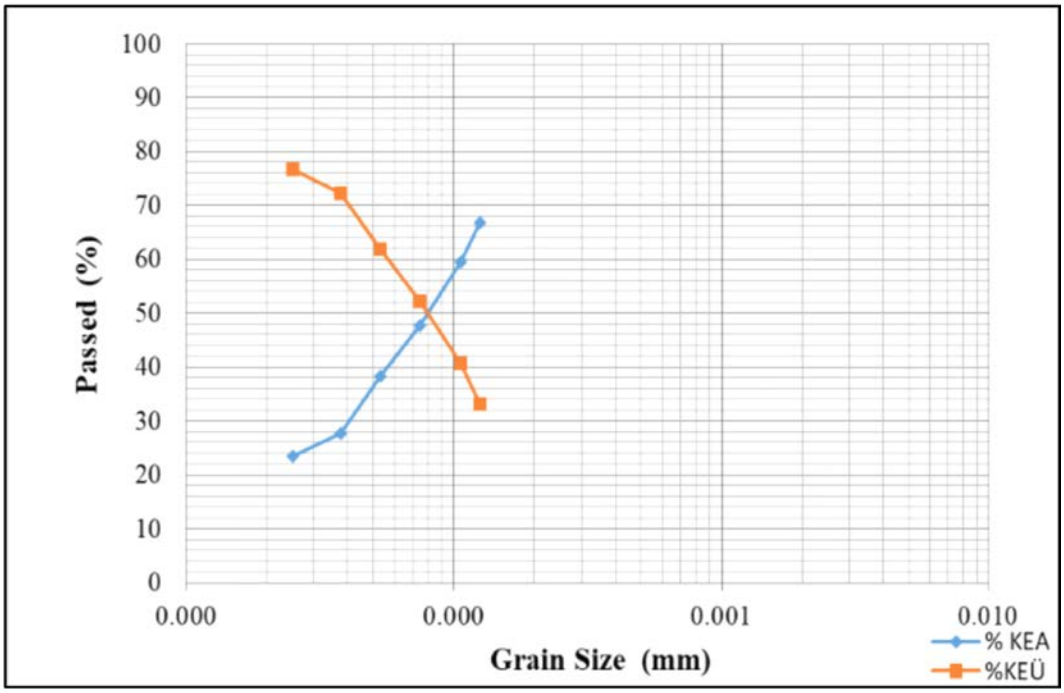

Figure 6. Sieve Analysis of C1 Sample

As seen in Figure 6, after the crushing process in the jaw crusher, the -2mm material is crushed in a ball mill for 20 minutes at 1000 rpm. subjected to the grinding process. At the end of the experiment, the cumulative under-sieve (CEA) D_{50} was found to be 0.08 mm and the cumulative over-sieve (KEU) D_{70} was 0.04 mm.

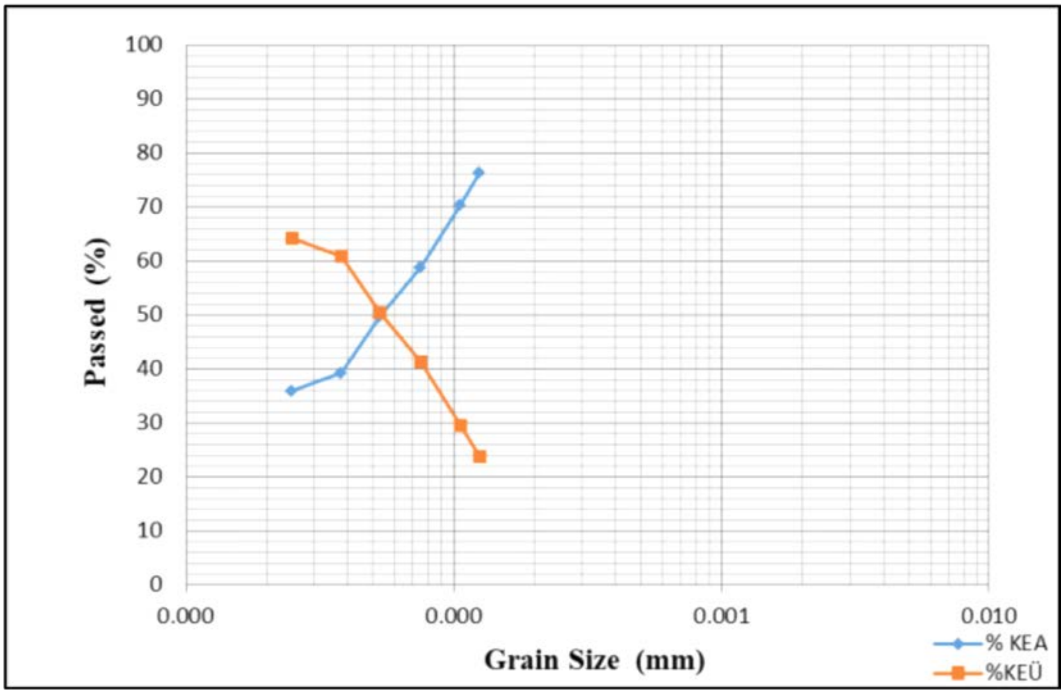

Figure 7. Sieve Analysis of C2 Sample

As seen in Figure 7, after the crushing process in the jaw crusher, the -2mm material is crushed in a ball mill for 30 minutes at 1000 rpm. subjected to the grinding process. At the end of the experiment, the cumulative under-sieve (CEA) D_{50} was found to be 0.05 mm and the cumulative over-sieve (KEU) D_{70} was found to be 0.1 mm.

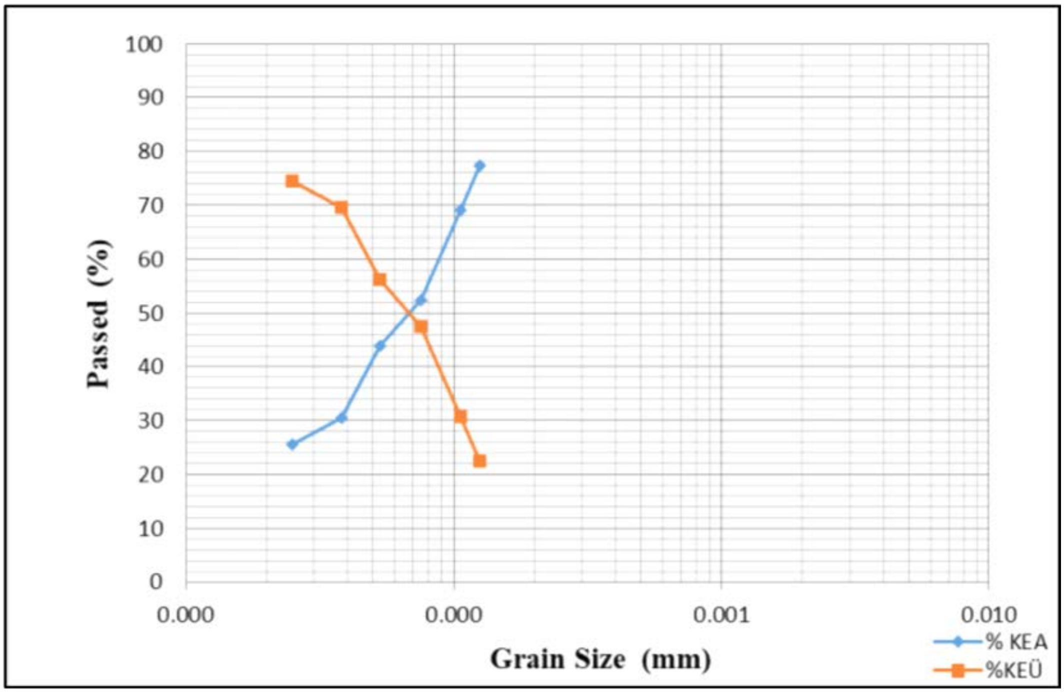

Figure 8. Sieve Analysis of C3 Sample

As seen in Figure 8, after the crushing process in the jaw crusher, the -2mm material is crushed in a ball mill for 40 minutes at 1000 rpm. subjected to the grinding process. At the end of the experiment, the cumulative under-sieve (CEA) D_{50} was found to be 0.07 mm and the cumulative over-sieve (KEU) D_{70} was found to be 0.04 mm.

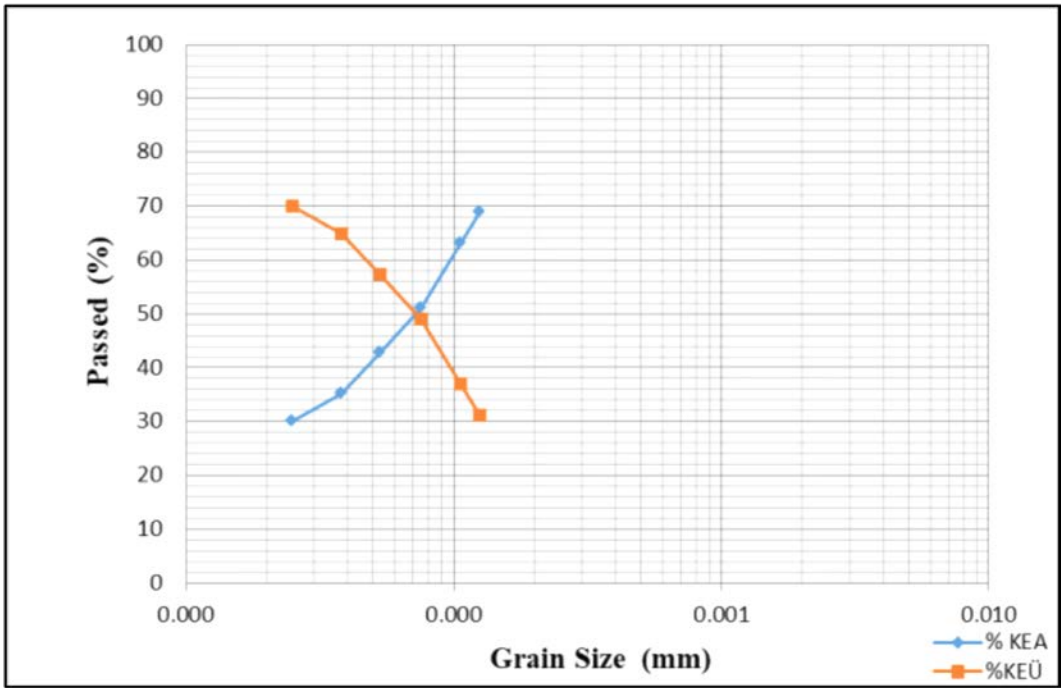

Figure 9. Sieve Analysis of C4 Sample

As seen in Figure 9, after the crushing process in the jaw crusher, the -2mm material is crushed in a ball mill for 60 minutes at 700 rpm. subjected to the grinding process. At the end of the experiment, the cumulative under-sieve (CEA) D_{50} was found to be 0.07 mm and the cumulative over-sieve (KEU) D_{70} was found to be 0.03 mm.

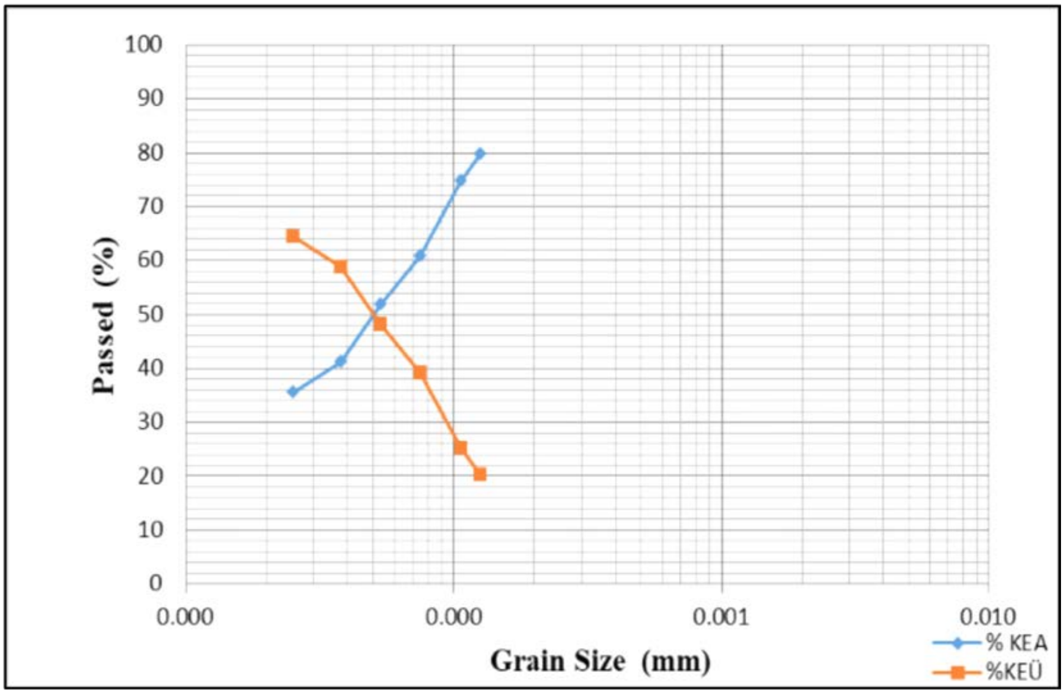

Figure 10. Sieve Analysis of C5 Sample

As seen in Figure 10, after the crushing process in the jaw crusher, the -2mm material is crushed in a ball mill for 90 minutes at 700 rpm. subjected to the grinding process. At the end of the experiment, the cumulative under-sieve (CEA) D_{50} was found to be 0.05 mm and the cumulative over-sieve (KEU) D_{70} was found to be 0.03 mm.

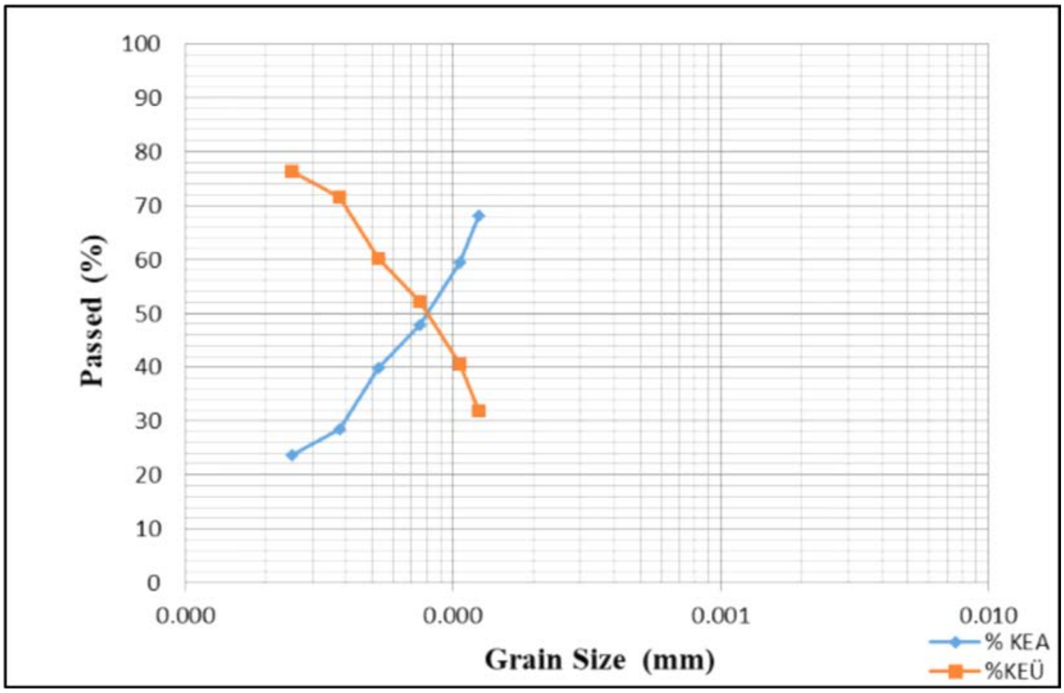

Figure 11. Sieve Analysis of C6 Sample

As seen in Figure 11, after the crushing process in the jaw crusher, the -2mm material is crushed in a ball mill for 120 minutes at 1250 rpm. subjected to the grinding process. At the end of the experiment, the cumulative under-sieve (CEA) D_{50} was found to be 0.08 mm and the cumulative over-sieve (KEU) D_{70} was found to be 0.04 mm.

As can be seen from the graphs above (Figure 6-11), the grain size generally decreases as the grinding time increases. One of the reasons for this is to increase the grinding speed. Since the change in the kinetic energy of the balls is directly proportional to the square of the grinding speed, the impact energy applied to the particles increased with ball-particle-ball collisions, and thus, smaller dimensional particles were obtained. As the ball-particle-weight ratio increases, there will be more balls in the grinding container, so more ball-particle-ball collisions will occur for the same grinding time, and thus, smaller powder size will be obtained as the grinded particles will interact with more balls for the same grinding time. The increase in grinding time, on the other hand, increased the number of these collisions as more time occurred for ball-particle-ball collisions and smaller particle size was obtained for longer periods until steady state conditions were reached.

3.3. Determination the Pozzolanic Activity of Basic Mineral Slag

Mortars were prepared using 0, 10, 20, 30 and 40% ground slag to be used in pozzolanic activity experiments. For each mortar group, 3 square prism samples of 40x40x160 mm were prepared (Figure 12). After the samples were prepared, they were kept on the drainage table for an average of 24 hours in order to drain the excess water in their bodies. At the end of this period, the samples were cured in the curing pool for 7/28 days in a way that the contact with the air was cut off.

Figure 12. Preparation of Prism Samples used in Flexural and Compressive Strength tests, curing of molds and samples removed from molds

3.4. Determination of Flexural and Compressive Strength of Mineral Slag

The bending test was carried out on beam samples of 4x4x16 cm in accordance with the principles specified in TS24 and TS-EN 196-1. Flexural strength tests were applied to 7 and 28 days old samples. The bending pressure press was adjusted in accordance with TS-3114 and automatically fractured, breaking loads and compressive stresses (bending and compressive strengths) were determined as shown in Figure-13.

Figure 13. Bending and Compressive Strength Tests on Beam Samples

The 7 and 28 day flexural and compressive strength values of the samples obtained by slag substitution at different rates are given in Table-7.

Table 7. 7 and 28 days flexural and compressive strengths of Samples at Different Mixing Ratios

Cement+Pozzolan (%)	**Compressive Strength (Mpa)**		**Flexural Strength (Mpa)**	
	7 Days	**28 Days**	**7 Days**	**28 Days**
0	3,10	3,73	1,11	1,15
10	2,42	3,59	1,03	1,20
20	1,98	2,98	0,92	1,05
30	1,25	2,43	1,00	0,99
40	1,25	1,73	1,00	0,96

It is expected that pozzolans will show a good reaction in a long time and gain a better internal structure and strength. Bending and compressive strength values of 7 and 28 days of prismatic samples poured using slag in different proportions were determined according to TS 24 and TS EN 196-1 (Figure 14 and Figure 15).

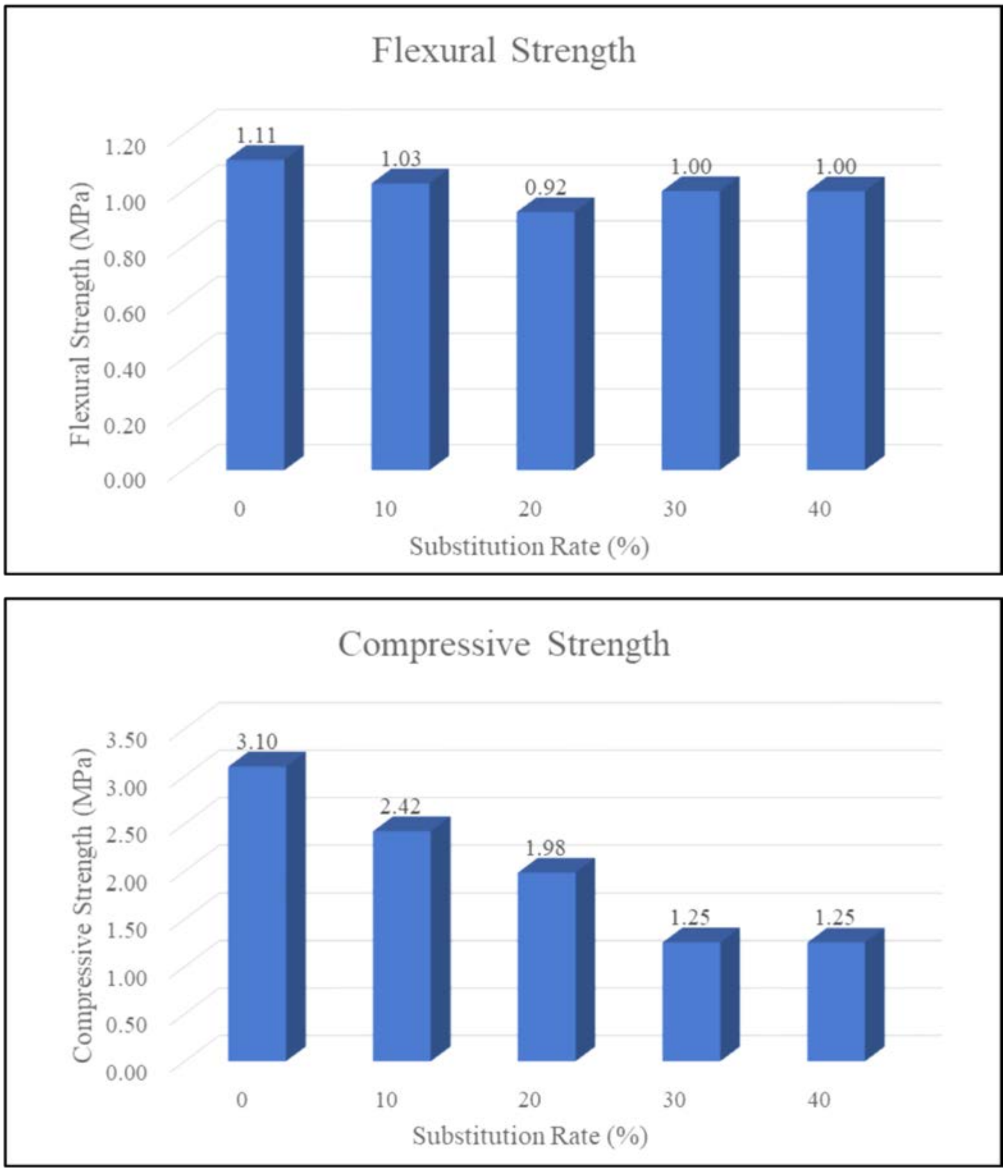

Figure 14. 7-day flexural and compressive strengths of the samples poured using slag in different proportions

As a result of the 7-day flexural strength tests, it was determined that the flexural strength of the samples with 20% replacement, where the flexural values of the slag substituted samples were approximately the same, decreased in flexural strength. In addition, it was determined that as the replacement slag ratio increased, the compressive strength decreased (Figure 14).

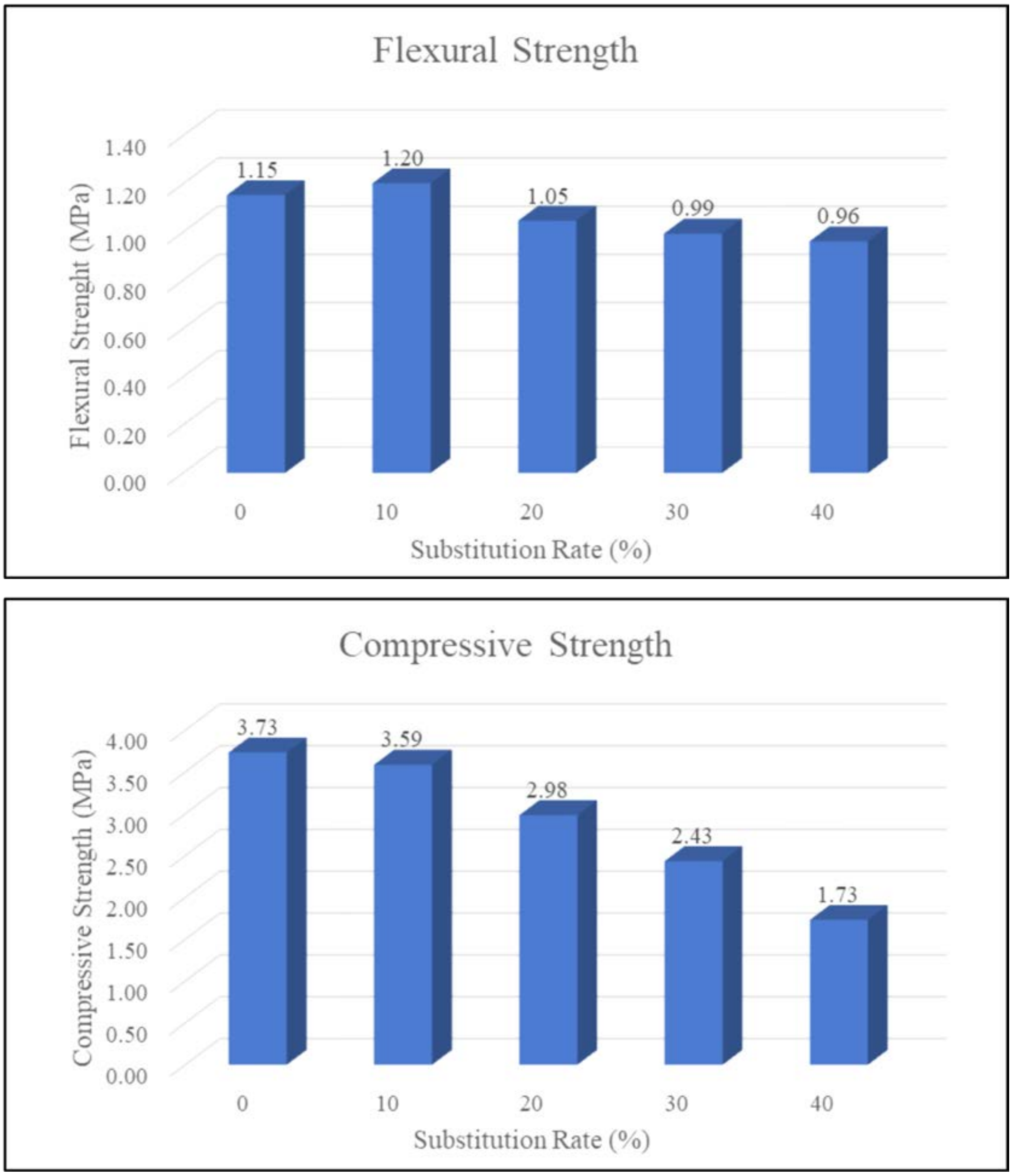

Figure 15. 28-day flexural and compressive strengths of the samples poured using slag in different proportions

As a result of the 28-day mechanical tests, it was observed that the flexural strength and compressive strength of the samples without slag (0%), with 10%, 20% replacement, increased compared to 7 days (Figure 15).The highest increase in compressive strength was observed in samples with 94.4% and 30% replacement.

4. CONCLUSION

As a result of the experiments carried out to determine the grinding parameters and pozzolanic activities of the old slags in the idle mining area in the Amasya region;

700 rpm. conditions are ineffective in particle size reduction and It was found that the increase in grinding time and ball powder weight ratios had little effect on particle size under low grinding speed conditions. The grinding speed is 1000 rpm. It is seen that the grinding reaches the optimal level and significant increases are seen in the specific surface area values,

When the size of the particles produced as a result of the mechanical grinding process is examined, the main mechanism affecting the size is the kinetic energy of the balls in the grinding container,

When the 7-day average bending and compressive strength values of slag+cement mixtures determined by pozzolanic activity tests are examined, increasing the amount of slag decreases both strengths,

The total SiO_2, Al_2O_3 and Fe_2O_3 contents of the slag used in the study are similar to pozzolanic materials such as baked clay and fly ash used in the cement industry, in accordance with the TS 25 limit values,

With the data obtained as a result of the study, it can be said that the production of cement with slag additives is promising in terms of low cost, energy efficiency and reduction in CO_2 emissions.

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