



EFFECT OF GRAPHITE POWDER ADDITIVES ON MECHANICAL PROPERTIES AND ELECTRICAL CONDUCTIVITY IN BLAST FURNACE SLAG-BASED ALKALI-ACTIVATED MORTARS

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Keywords

*Alkali-Activated Mortars,
Graphite,
Compressive Strength,
Flexural Strength,
Electrical Conductivity.*

Abstract

In this study, the effect of graphite powder additive on mechanical properties and electrical conductivity of alkali-activated mortar samples produced using blast furnace slag was investigated. In the preparation of the mortar samples, graphite powder in (<75) micron size was substituted at a rate of 0%-0.5-1%, 2% and 4% by weight of the binder. Sodium hydroxide and sodium silicate were used as activators in the mortar samples produced with Blast Furnace slag, and the samples were thermal cured at 110°C for 24 hours. Workability, unit weight, electrical conductivity, tendencies and compressive strength of all mortar samples that completed the curing period were determined. In addition, experiments were carried out to determine the water absorption and void ratios of the samples that gave the best results in the cementitious system activated with alkalis. The results obtained showed that the workability of the graphite powder was improved at 1% reinforcement rate in the mortar samples activated with alkalis, and it had a negative effect at the rates above 1%. It was understood that 1% graphite powder additive contributed positively to flexural and compressive strengths, while 4% graphite powder additive contributed provided the highest electrical conductivity.

YÜKSEK FIRIN CÜRUFU TEMELLİ ALKALİLERLE AKTİVİTE EDİLMİŞ HARÇLARDA GRAFİT TOZU KATKISININ MEKANİK ÖZELLİKLER VE ELEKTRİKSEL İLETKENLİK ÜZERİNDEKİ ETKİSİ

Anahtar Kelimeler

*Alkalilerle Aktivite Edilmiş
Harçlar,
Grafit,
Mekanik Dayanım,
Eğilme Dayanımı,
Elektriksel İletkenlik.*

Öz

Bu çalışmada yüksek fırın cürufu kullanılarak üretilen alkalilerle aktivite edilmiş harç numunelerde grafit tozu katkısının mekanik özellikler ve elektrik iletkenliğine etkisi araştırılmıştır. Harç numunelerin hazırlanmasında bağlayıcı oranın ağırlıkça %0-%0,5-%1-%2 ve %4 oranında, (<75) mikron boyutunda grafit tozu ikame edilmiştir. Yüksek Fırın cürufu ile üretilen harç numunelerde aktivatör olarak sodyum hidroksit ve sodyum silikat kullanılmış ve numuneler 24 saat 110°C'de ısı küre tabi tutulmuştur. Kür süresini tamamlayan tüm harç numunelerin işlenebilirlik, birim ağırlık, elektriksel iletkenlik, eğilme ve basınç dayanımları belirlenmiştir. Ayrıca, alkalilerle aktivite edilmiş çimentolu sistemin de en iyi sonuçları veren numunelerin su emme ve boşluk oranlarını belirleyen deneyler yapılmıştır. Elde edilen sonuçlar; grafit tozunun alkalilerle aktivite edilmiş harç numunelerde %1 takviye oranında işlenebilirliğin iyileştiği, %1'in üzerindeki oranlarda olumsuz etki yaptığı görülmüştür. %1 grafit tozu katkısı eğilme ve basınç dayanımlarına pozitif katkı sağlarken, %4 grafit tozu takviyesi ise en yüksek elektriksel iletkenliği sağladığı anlaşılmıştır.

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Highlights

- Investigation of the impact of graphite powder additives on the mechanical properties and electrical conductivity of alkali-activated mortars with blast furnace slag.
- Evaluation of flow amount, workability, compression strength, electrical resistivity, and conductivity of the samples.
- Findings indicate that the addition of graphite enhances the workability and increases the flexural and compression strengths at lower ratios, while higher ratios lead to a decrease in mechanical strength.
- Graphite additives provide the mortars with electrical conductivity, with higher graphite ratios resulting in increased conductivity.
- The study showcases the possibility of creating conductive structures by incorporating graphite into alkali-activated mortars, which expands their potential engineering applications.

Graphical Abstract

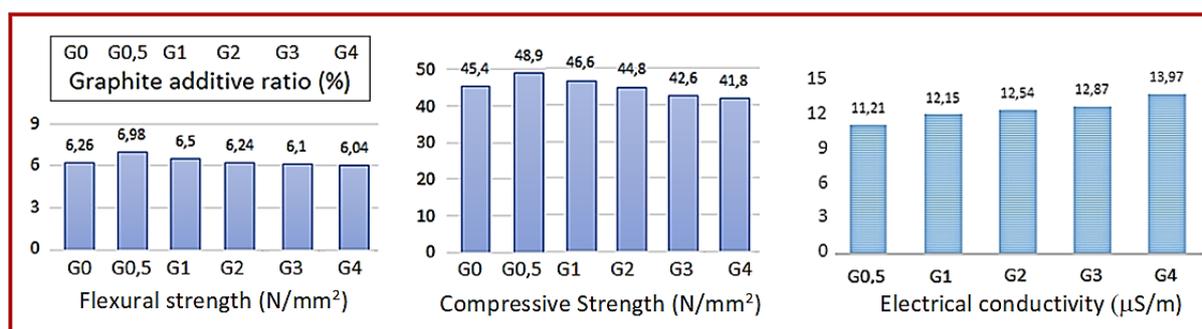


Figure. Mechanical strength and electrical conductivity of graphite added mortar

Purpose and Scope

In this study, it is aimed to examine the effect of graphite, which is a carbon allotrope and a good conductor, on the electrical properties of the mortar along with its mechanical properties.

Design/methodology/approach

For this purpose, test samples were prepared by adding graphite powder at different replacement ratios into the mortar. In the first stage, the flow amount, flexural and compression strength tests were carried out on the samples. Then, electrical resistivity and conductivity measurements were carried out. The findings were evaluated by creating graphics.

Findings

It has been determined that the graphite added to the mortar increases the workability, increases the flexural and compression strengths at 0.5% and 1% rates, but decreases the mechanical strength at higher rates. It has been observed that the graphite added to the mortar gives the samples electrical conductivity properties and this conductivity value increases depending on the increase in the graphite ratio.

Originality

With this study, it has been seen that the concrete mortar, which is an insulating composite, has a structure that can conduct electricity with the addition of graphite. Depending on the graphite ratio in the structure and the production technique, it is considered that such composite structures may be used for different engineering applications where certain levels of conductivity are required and will encourage more extensive research.

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1. Introduction

The industry with the highest carbon dioxide emission is cement production facilities. Very high levels of carbon dioxide are emitted to produce 1000 kg of cement (Hasanbeigi *et al.*, 2010). The amount of carbon dioxide from cement production constitutes 6 percent of the total carbon dioxide (Chen *et al.*, 2010).

Cement producing facilities produce approximately 1.5 billion tons each year and this production continues to increase every year. Clinker grinding constitutes approximately 40 percent of cement production. This grinding process requires a lot of electrical energy (Jankovic *et al.*, 2004). For this reason, various studies are carried out to develop alternative binders to today's cement systems. In this context, one of the alternative binders studied is concrete activated with alkalis. These concretes are green building materials that will replace cement and be produced such as fly ash or blast furnace slag to be used with waste slags (Amer *et al.*, 2021; Sandanayake *et al.*, 2018).

In a recent study by (Huseien, 2023), various nanomaterials were investigated for their potential in producing high-performance cementitious, geopolymer, and alkali-activated concrete composites. The study analyzed the effects of these nanomaterials on the fresh properties, mechanical properties, and durability of the modified concrete composites. The findings highlighted the improvements in microstructure and mechanical properties, such as anti-wear and anti-chloride penetration, achieved through the use of nanomaterials like SiO₂, Al₂O₃, TiO₂, and Fe₂O₃.

In another study, (Matalkah & Soroushian, 2020) focused on the effects of incorporating graphene nanoplatelets (GNPs) into alkali-activated binders. The research examined the impact of GNPs on the microstructure, heat of hydration, mechanical properties, and durability characteristics of the concrete. The inclusion of GNPs was found to enhance abrasion resistance, moisture sorptivity, and the formation of hydration products. Good bonding between GNPs and the alkali-activated matrix was also observed, leading to improved performance.

To gain the conductive properties of concrete, conductive materials must be added. Depending on the properties of the conductive material, different properties may develop in the concrete. It is possible to use it in different applications according to its features such as snow melting (Tumidajski *et al.*, 2003), increased cathodic protection (Sun *et al.*, 2021), energy discharge electrodes (Zhang *et al.*, 2017) or blocking or increasing signal transmission (Jung *et al.*, 2020). As the concrete gains a conductive property, it can also be used for traffic and health services. The increase in the electrical conductivity of the material causes a decrease in the resistivity at the same rate (El-Dieb *et al.*, 2018; Haddad & Chung, 2017; Zhang *et al.*, 2017). Graphite powder conducts electricity well and is cheap in price. For this reason; It is a good filling element used in fields such as medicine, engineering and science (Anwar *et al.*, 2014; Peyvandi *et al.*, 2013). Graphite powder shows much better conductivity than copper. Because of this feature; it used as a filler to composite materials, engines or mortars (Uysal, 2012). The higher the value of the current flowing through the new material produced, in other words, the lower the resistance of the material, the higher its electrical conductivity (Costa & Henry, 2011). The electrical conductivity was investigated by adding fly ash, silica fume and blast furnace slag to the setting cement paste. It was observed that the cement paste hardened with the increase of added minerals. Since this hardening prevents the permeability of electric current, the electrical conductivity has decreased (Topcu *et al.*, 2012). Investigation of electrical resistivity of blast furnace slag added cement pastes was investigated. In all water/cement ratios, according to the hydration time, as the YFC ratio in the mixture increased, the electrical current transmission decreased and the electrical resistivity increased. This is due to the fact that foreign materials such as silica and alumina in the content of YFC conduct the electric current, but when more than 10% of YFC is used, it is thought that the calcium hydroxide (Ca(OH)₂) produced by the hydration of Portland cement is not enough (Topçu *et al.*, 2018). The electrical conductivity, diffusion and permeability of Portland's cement based mortars were investigated. The electrical conductivity of a series of cementitious binders was studied over 450 days. Bulk conductivity measurements on mortars and related pore fluids were investigated. It is showed that over the first 28 days of hydration, changes in pore structure had a greater effect on the measured conductivity than changes in pore fluid conductivity (McCarter *et al.*, 2000). The electrical properties of Portland cement-silica fume and calcium hydroxide-silica fume pastes were investigated. It has been observed that the paste is affected by the molar Ca(OH)₂/silica ratio, where the conductivity is most intense. While the conductivity has the lowest siemens value at Ca(OH)₂/silica = 0.80, the conductivity has the highest density at Ca(OH)₂/silica = 1.0. Therefore, the molar ratio of Ca(OH)₂/silica of 1.0 was found to represent the maximum initial hydrolysis rate of the Ca(OH)₂-SF mixture (Salem, 2002).

Today, it is very important for sustainability to transform wastes into a new material by using them as raw materials. An example of this is the production of alkali-activated mortar or concrete with alternative binders for cement systems. In this study; Unlike the literature, it is aimed to examine the electrical conductivity together with some mechanical properties by producing mortars that are activated with high alkalis and add graphite powder.

2. Material and Method

2.1. Material

The sand added to the cement for this article was taken from the Limak Trakya Cement Factory. The type of this sand is CEN sand determined according to TS EN 196-1 standards (Standard, 2006). Standard Rilem sand, typically has a specific gravity of 2.63 g/cm^3 and a unit weight of $1600\text{-}1700 \text{ kg/m}^3$. The blast furnace slag used was produced in the Ereğli Iron and Steel Factory. The grain distribution of the reference sand is given in Table 1, the chemical and physical properties of the blast furnace slag are given in Table 2, and the properties of the additive element graphite are given in Table 3. Blast furnace slag was used directly without grinding as it left the factory.

Table 1. Grain distribution of the reference sand

Sieve sizes (mm)	Cumulative Sieve Remainder (%)
2.00	0
1.60	7 ± 5
1.00	33 ± 5
0.50	67 ± 5
0.16	87 ± 5
0.08	99 ± 1

Table 2. Blast furnace slag technical specifications

Chemical Property (%)	Blast furnace slag
SiO ₂	35.2
Al ₂ O ₃	17.51
Fe ₂ O ₃	0.68
CaO	37.7
MgO	5.51
SO ₃	0.69
Na ₂ O	0.42
K ₂ O	1.71
Physical property	
Blaine fineness (cm ² /g)	3940
Specific Gravity (g/cm ³)	2.89

Table 3 Graphite powder specifications

Physical property	
Chemical formula	C
Powder size (μm)	μ75
Specific Gravity (g/cm ³)	2.1
Colour	Carbon black
Thermal conductivity (W/mK)	In X-Y axis: 1200-1400
Thermal conductivity (W/mK)	In Z axis: 10-15
Electrical Conductivity (S/m)	130
Melting point (°C)	3650
Compressive strength (MPa)	25-190
Flexural strength (MPa)	10-100

Digital camera image, microstructural image under scanning electron microscope (SEM) and graphite powder of blast furnace slag used in the production of test sample mortars are shown in Figure 1.

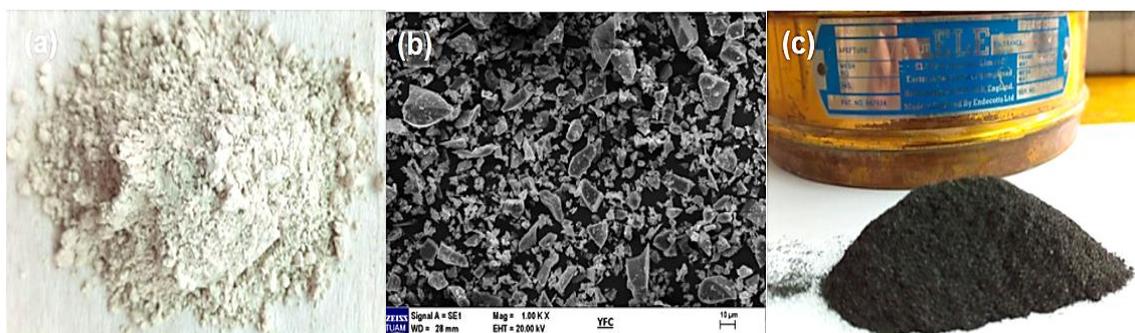


Figure 1. Blast furnace slag (a) digital camera ,(b) SEM image, (c) graphite powder

In the experimental study, solid sodium hydroxide (NaOH) and sodium silicate solution (Na₂SiO₃) with 98% purity were used as alkali activators. The chemical composition of the Na₂SiO₃ solution (wt%): SiO₂ (26.5), Na₂O (8.3) and dH₂O (6.2). In the experimental study, alkali activators were utilized to enhance the reactivity and accelerate the reaction rate. Solid sodium hydroxide (NaOH) and sodium silicate solution (Na₂SiO₃) with a purity of 98% were employed as the alkali activators. Activators are substances that are employed to increase or initiate the reaction rate. In this context, they play a crucial role in the alkali activation process by promoting the desired chemical reactions. In the production of concrete and mortar, alkali activators are used to expedite the hydration process and facilitate the rapid hardening of the material. This leads to the development of early strength and improved mechanical properties. Specifically, in this study, solid sodium hydroxide (NaOH) and sodium silicate solution (Na₂SiO₃) were utilized as alkali activators. Sodium hydroxide, when dissolved in the concrete or mortar mixture, releases hydroxide ions (OH⁻) and initiates the alkali activation process. On the other hand, sodium silicate solution consists of sodium oxide (Na₂O) and silica (SiO₂). This solution is employed to initiate hydration reactions and accelerate the activation process. The use of activators allows concrete and mortar materials to gain early strength and become ready for use in a shorter period. Furthermore, activators contribute to improving the mechanical properties and durability of the material. Hence, activators play a vital role in the alkali activation process, aiding in achieving the desired properties and enhancing the overall performance of the material. Sodium hydroxide and sodium silicate used as activators are shown in Figure 2.

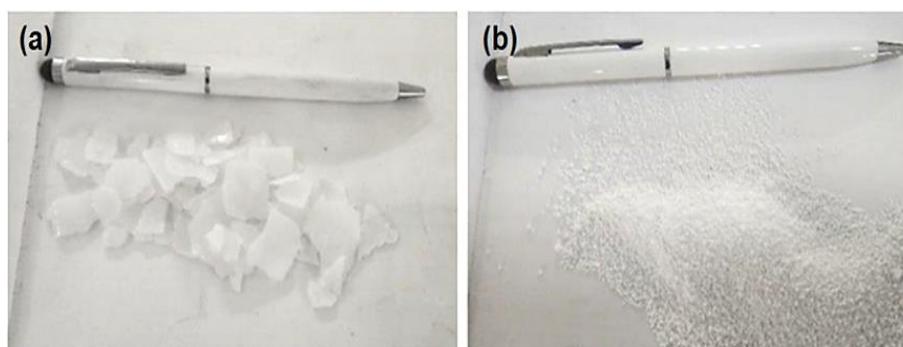


Figure 2. Sodium hydroxide (a) and sodium silicate (b)

Potable tap water was used in the experimental study.

2.2. Experiment Design

The mixing ratios of the alkali-activated mortars produced in this experimental study are given in Table 4.

Table 4. Mortar mixing ratios

Sample	BFS (g)	NaOH (g)	Na ₂ SiO ₃ (g)	Sand (g)	Water (g)	Graphite (g)
GO Pure	450	32	127	1350	146	0
G0.5	447.75	32	127	1350	146	2,25
G1	445.5	32	127	1350	146	4,5
G2	441	32	127	1350	146	9
G3	436.5	32	127	1350	146	13,5
G4	432	32	127	1350	146	18

In the first stage of the experimental study, workability tests of the produced mortars were carried out. The images of the fresh mortars from the Fresh Mortar Consistency Determination experiments are shown in Figure 3. These tests were carried out in the flow table according to TS EN 1015-3 (TS ENV1317-4, 2011).



Figure 3. Flow view of mortar samples

2.3. Mechanical Experiments

In this part of the article, the mechanical properties of the mortars of the filling material are examined. The mortars formed were prepared in dimensions of 40×40×160 mm. Materials were produced in equal dimensions for the application of flexural and compressive strength tests. These tests were carried out according to TS EN 196-1 standards. The prisms obtained from the specimens subjected to the flexural test were placed in such a way that they did not exceed the plates of the test set by ± 5 mm. Experiment set for compressive strength, It has been adjusted to not exceed the speed range of 2200 N/s and 2600 N/s. This device was loaded until the prism broke. Compressive strength was obtained by taking the arithmetic average of the pressure tests of six mortars determined from three prismatic sets. More than $\pm 10\%$ discarded this result because one of the six results differed from the mean. The remaining five results were averaged. The results obtained for flexural and compressive strength tests are shown in Figure 4.

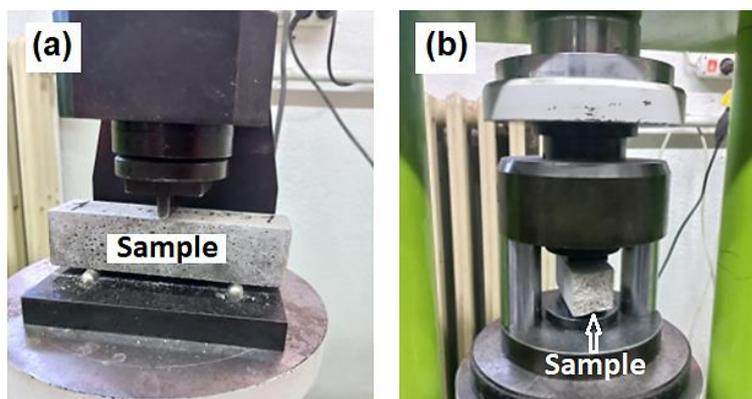


Figure 4. Flexural strength test (a) Compressive strength test (b)

2.4. Electrical Conductivity Test

The experimental setup used in the electrical resistivity measurement is shown in Figure 5. A DC power source and two digital multimeters were used in the established measurement setup. The DC power supply is used to give a constant DC voltage to the material produced. Digital multimeters are used to measure the electric current passing through the material and the voltage falling on it. The currents passing over it were measured by fixing the power supply at 30V. While measuring these currents, measurements were taken in different parts of the material. The average value was calculated by taking measurements from 5 different places of the produced material. The reason for this is that the material produced is not homogeneous. It was tried to calculate the optimum current value for the material by taking measurements from different parts.

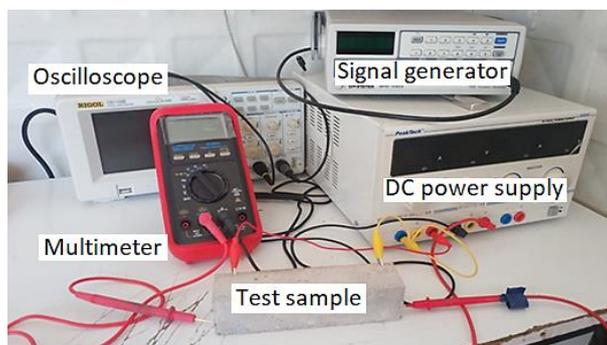


Figure 5. Experiment setup used in electrical resistivity measurement

According to the current value obtained, the resistance values of the material to the voltage values given from the DC power source were calculated. The resistance value of the material was calculated using the expression in Equation 1. In Equation 1; R value is resistance (Ω), I value is current (A) and V value is voltage (V).

$$R = \frac{V}{I} \tag{1}$$

The resistivity value of the material with the resistance value was calculated. However, as the size and area of the material change, the resistance value changes. If the size of the material differs, the results may differ as the diffusion of graphite powder in it will also differ. For this reason, 40x40x160 mm dimensions are used in every material produced. Thus, measurements were taken under equal conditions for each material produced. In Equation 2, the formula that gives the resistance value according to the size and volume of the material is shown. The resistivity (ρ) is drawn from this formula and shown in equation 3. In Equations 2 and 3; L value gives the length of the material (m), A value gives the area of the material (m^2) and ρ gives the resistivity of the material ($\Omega.m$) (Heinzel vd, 2004).

$$R = \frac{L \cdot \rho}{A} \tag{2}$$

$$\rho = \frac{R \cdot A}{L} \tag{3}$$

The electrical conductivity expression of the material after determining the resistivity is also shown in equation 4. The σ value represents the electrical conductivity (S.m).

$$\sigma = \frac{1}{\rho} \tag{4}$$

3. Results & Discussion

3.1 Unit weight

The graph created according to the unit weights obtained from the graphite added mortar samples is given in Figure 6.

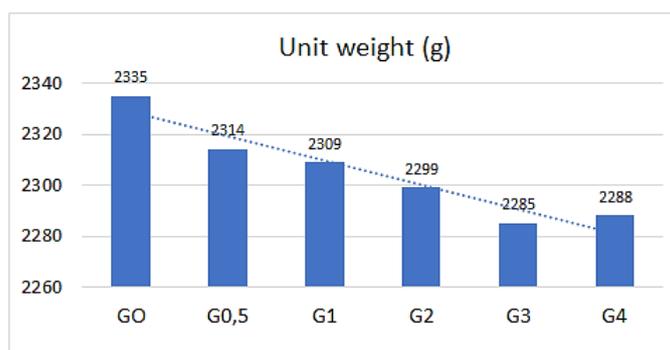


Figure 6. Unit weights of mortar samples

According to the results, the unit weight values of the samples produced with graphite powder substitution decreased compared to the sample without graphite additive. While the highest unit weight values were in the pure control sample, the highest unit weight in the graphite powder added samples was obtained in the samples with the lowest additive rate of 0.5% graphite powder. On the other hand, the amount of activator used in the preparation of the mortars did not make a significant difference in the unit weight values of the samples. The reason for this is that the addition of graphite powder to the mortar caused a decrease in unit weight values compared to the sample without graphite additive. This decline can be attributed to several factors. First, graphite has a lower density compared to other materials typically used in mortar mixes. This lower density contributes to a reduction in the overall weight of the mortar and leads to a reduction in unit weight. Additionally, the presence of graphite particles in the mortar mixture fills the void spaces between the aggregate particles and the binding material. This filling effect reduces the overall volume of the mortar, resulting in a decrease in its unit weight. Moreover, graphite powder has a relatively low specific gravity, meaning that it weighs less for a given volume compared to other components of the mortar mixture. As a result, the inclusion of graphite powder in the mixture further contributes to a decrease in the overall weight of the mortar, leading to a decrease in its unit weight. It is worth noting that the amount of activator used in the preparation of the mortars did not significantly affect the unit weight values. This suggests that the observed decrease in unit weight is primarily influenced by the addition of graphite powder rather than the activator content.

3.2 Flow-Table Experiment

The spread rates obtained as a result of the flow tests of the graphite-added mortar samples and the deviation rates of the spread values compared to the non-graphite-added sample are shown in Figure 7.

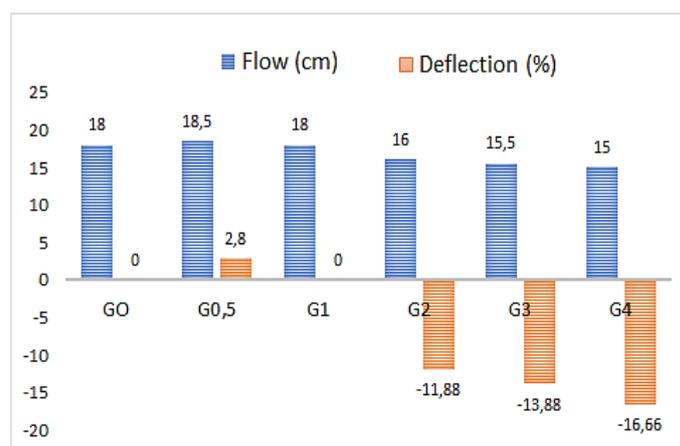


Figure 7. Flow diameter

It can be seen in Figure 7 that the 0.5% graphite additive added to the sample increases the flow value and thus the workability by 2.80%. After the addition of 1% graphite powder, the flow amount of the mortar samples decreased and was adversely affected. In a similar study in the literature, in their study (Wang et al., 2016), It has been observed that graphene oxide, a derivative of graphite, improves the hydration of alkali-activated and reduces the amount of water in the alkali-activated structure. This indicates that the alkali-activated mortar reduces its fluidity. (Li et al., 2017) showed that the reduction of free water in the alkali-activated mortar resulted in higher water holding capacity of graphene oxide aggregates formed by chemical crosslinking of graphene oxide nanoplates with calcium ions. It is stated that this situation causes the alkali-activated mortar to decrease its fluidity. In another research results, it was observed that graphene oxide mixed with alkali-activated mortar reduced the fluidity of the alkali-activated mortar and the slump of concrete (Lu & Ouyang, 2017).

3.3 Evaluation of Flexural and Compressive Strength Results

Graphs showing the flexural and compressive strength of graphite powder substituted mortar samples at 0% - 0.5% - 1% - 2% - 3% and 4% weight ratios are given in Figure 8 and Figure 9. In addition to the results; the differences in the strength values of the graphite added mortars compared to the graphite-free sample are also shown in the graphs as percentage deviations

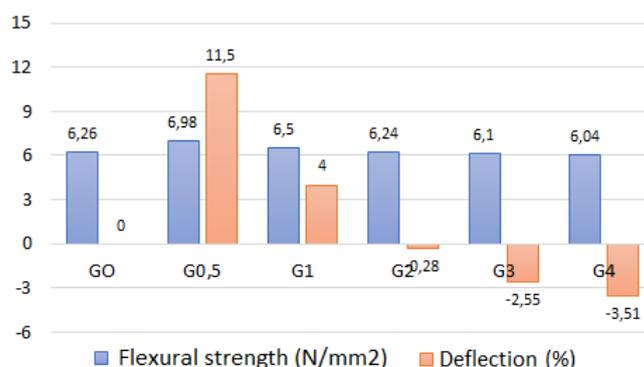


Figure 8. Flexural strength values

The observed increase in flexural strength values with the addition of 0.5% and 1% graphite powder, as shown in Figure 8, suggests that graphite powder can have a positive effect on the flexural strength of the mortar. However, it is important to note that a decrease in flexural strength is observed after 1% graphite powder addition. This decrease in strength can be attributed to the low adhesion ratio between the graphite particles and the material. The bonding force between the graphite particles and the matrix material plays a significant role in determining the compressive strength of the mortar. When the adhesion ratio is low, the bonding between the graphite particles and the matrix becomes weaker, leading to a decrease in compressive strength. This finding is consistent with the research by (El-Dieb et al., 2018) which emphasizes the influence of the bonding force on the resulting compressive strength. It is important to consider that the effect of graphite powder on strength is not solely determined by its adhesion characteristics. Other factors such as the distribution of graphite particles within the mortar matrix and the potential formation of weak interfaces can also contribute to the observed decrease in flexural strength after a certain graphite powder content. The decrease in flexural strength after 1% graphite powder addition can be attributed to the low adhesion ratio between the graphite particles and the material, which affects the bonding force and consequently influences the compressive strength.

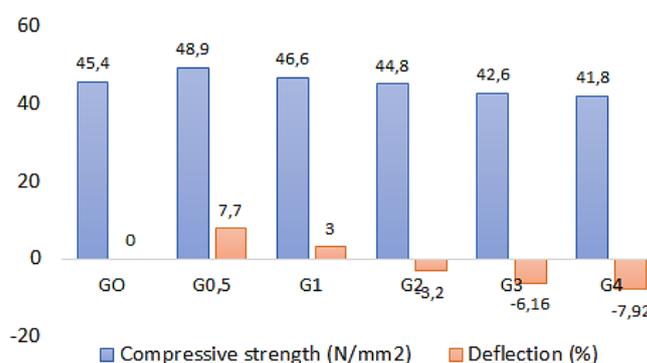


Figure 9. Compressive strength values

When the compressive strength was compared with the sample without graphite additive, it was observed that it increased by 7.70% in the sample with 0.5% graphite powder and 3% in the sample with 1% graphite powder. However, there was a decrease in compressive strength after 1%. In general, the flexural and compressive strengths of cementitious materials are related to the relatively weak bond between the graphite particles and the matrix phase of the mortars. It is clearly seen that the strength decreases linearly with the addition of graphite powder. Moreover, these mechanical strength results can be attributed to the weaker bond between the matrix of the mortars and the hydrophobic graphite particles and the layered structure of the graphite, which can act as a stress concentrator for crack propagation. According to studies in the literature, it has been observed that graphite powder has a negative effect on the mechanical performance of electrically conductive cementitious composites. The mechanical improvement observed with the inclusion of slag in this structure affects the crystal structures, which strengthens the bond between the aggregates and the C-S-H gel (Abedini & Zhang, 2021; Mou et al., 2019).

3.3 Electrical Resistivity Tests and Evaluation of Results

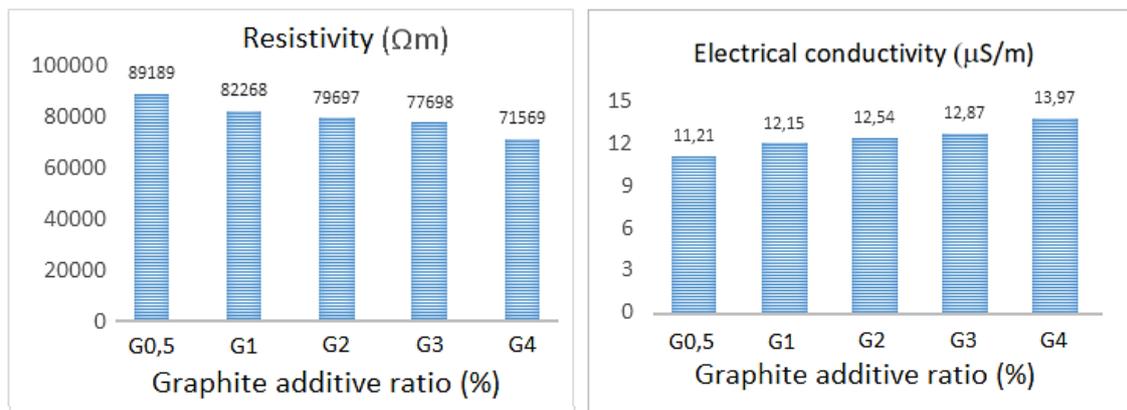


Figure 10. Resistivity and electrical conductivity values of mortar samples

The resistivity and electrical conductivity values obtained by adding different graphite ratios to the samples are shown in Figure 10. When the resistivity and electrical conductivity graphs obtained in Figure 10 are examined, The electrical conductivity of the sample with G4 additive increased by 24.6% compared to the sample with G0.5 additive. The electrical conductivity of the new material was increased with the addition of graphite. This ratio also showed that the graphite was homogeneously dispersed in the produced mortar. It has been observed that the positive effect on the material in terms of conductivity increases as the density of graphite is increased. Since graphite is a very good conductor, it is expected that the resistance to electric current will decrease. The decrease in the resistivity value caused the electrical conductivity value to increase at the same rate.

4. Conclusions

An increase of approximately 12.8% in flow diameter was observed in the mortar samples containing 1% graphite powder based on blast furnace slag activated with alkalis. However, after adding 1%, the workability decreased. The addition of graphite to the mortars caused a decrease in the flexural and compressive strengths mainly due to the increased demand for mixing water and the resulting increased porosity of the mortar. While the reduction in fluidity can be a significant barrier to the in situ use of this composite material; Reductions in compressive strength may prevent its use in structural applications. It was also understood that graphite does not affect the hydration of alkali-activated mortar, that is, it functions as an inert conductive filler. With the addition of graphite, the mortar showed electrical conductivity and this conductivity value increased due to the increase in the graphite ratio. However, the use of graphite as a conductive additive in alkali-activated mortar has shown that it may bring some practical limitations. In conclusion; AAC composites produced as conductors for a variety of applications will have good potential for structural health monitoring systems for highway bridge decks, parking lots, sidewalks, driveways and airport runways, coated snowmelt systems, smart structures.

Conflict of Interest

No conflict of interest was declared by the authors.

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