

Investigation of the Physical and Mechanical Properties of Metabentonite and Slag-based Geopolymer Mortars

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ABSTRACT

With population growth and industrialization, fly ash accumulates in ground blast furnace slag and other industrial waste landfills. Since these wastes are the main source of pollution, they are harmful to the environment, but in the production of geopolymer mortar or concrete, these solid wastes are recycled and added to the system. In this study, metabentonite (MB)-slag-based geopolymer mortars, which are formed by calcining bentonite clay with by-products such as blast furnace slag, were prepared. In this context, bentonite was transformed into metabentonite by calcining at 900 °C. In the study, geopolymer mortars based on metabentonite-ground granulated blast furnace slag (100 MB, 75 MB+25 GGBFS, 50 MB+50 GGBFS, 25 MB+75 GGBFS) and containing recycling aggregate as fine aggregate was produced in five series and physical (unit weight, water absorption and void ratio) and mechanical tests (compressive and flexural strength, ultrasonic pulse velocity) were carried out on the 7th and 28th days. The conclusions showed that although the highest strength was obtained in the series containing 75 GGBFS+25 MB, remarkable physical and mechanical properties were obtained for the series containing 75 MB+ 25 GGBFS in both molar concentrations, and an environmentally friendly and economical geopolymer composite design could be formed.

Keywords: Geopolymer, bentonite, physical properties, mechanical properties, sustainability.

INTRODUCTION

The term geopolymer was originally used in 1978 by Davidovits J. In structures formed by inorganic molecules. It is also stable up to 1250 °C, but is non-flammable. It may become polycondat due to activation with an alkaline solution at temperatures lower than 100 °C. Geopolymerization can be expressed by the formation of covalently bonded compounds by sharing the electrons of the oxygen atom of the eliminates and silicates in the structure of the baked clay. In the structure of these materials, most natural heat-activated silicon and alumina minerals (aluminum and silicon oxide compounds) are alkaline-activated and strengthen intermolecular bonds, thus forming polymers. Geopolymers can be of crystalline or amorphous structure, and they are similar to natural rocks in terms of properties. Cement obtained in this manner is referred to as alkali-activated cement or inorganic polymer cement. The geopolymer process contains above all mineral molecules such as Si, Al, Mg,

Ca, P, K, and N. It is formed with the formation of covalent chain polymers formed following the polycondensation of monomers between them. The products obtained by firing the solution obtained by activating the metakaolin clay calcined at the 750 °C with alkali salt and alkali silicates at low temperatures (40-100 °C) are the conditions in which the geopolymer material shows the best properties. At the same time, these materials are known as new-generation inorganic binders with high potential, replacing Portland cement-based mortars (Shaikh, 2016; Maholtra, 2002; Madloul et al., 2011; Al Bakri et al., 2011; Aygörmez, 2018; Hassan et al., 2019; Beycioğlu et al., 2008; Bilgen et al., 2011). It is known that fly ash, ground granulated blast furnace slag (GGBFS), rice husk ash and other industrial wastes accumulate in landfills, especially with population growth and industrialization, and these wastes are the main source of pollution. The production of geopolymers is an environmentally friendly construction and building material based on the recycling of these industrial solid

RESEARCH ARTICLE
 PII: S225204302300005-13
 Received: July 11, 2023
 Revised: September 16, 2023
 Accepted: September 17, 2023

wastes mentioned in the text. For example, GGBFS is a high-temperature by-product during the extraction of iron ore and consists mainly of lime and Ca-Mg alumina silicates. The high glassy and granular content of the GGBFS increases the reactivity of the mixture, thereby accelerating the polymerization process. Some researchers have used GGBFS as an additive to increase the strength of geopolymer mortar or concrete. It has been observed that these materials exhibit less machinability due to their irregular particle shapes. A higher GGBFS percentage accelerated the initial and final hardening time of the geopolymer mixture and increased the compressive strength. This may lead to the conclusion that GGBFS can also promote durability as a structural repair material. Bentonite, another exemplary material apart from these, is a clay mineral consisting mainly of montmorillonite, which is formed as a result of the chemical weathering of volcanic ash, tuff, and lava. Turkey has approximately 250 million bentonite reserves and the largest bentonite deposits are in the areas of Ankara, Çankırı, Ordu, Trabzon, Elazığ, Malatya and Bartın. The use of bentonite in a variety of industrial processes is a direct function of its structure and composition. Grain size and shape, surface chemistry and characteristics such as surface abrasion, viscosity, absorption, and adsorption significantly affect its use in various domains. Bentonites are classified into 3 different groups: calcium bentonite which does not swell in water, sodium bentonite which swells in water, and balanced sodium-calcium-bentonite, which has less of these properties. While sodium bentonite swells from 8 to 10 times its own volume in contact with water, the swelling rate of calcium bentonite is approximately 2 to 3 times. [Mirza et al. \(2009\)](#) investigated the effect of Pakistani bentonite in mortar as a low-cost construction material. In the study, bentonite and calcined bentonite (metabentonite) (150 °C, 250 °C, 500 °C, 750 °C and 950 °C) were evaluated replacement of traditional Portland cement. Results demonstrated that 20% of the non-calcined bentonite and 25% of the bentonite calcined at 150 °C can be used as a low-cost construction material in the mortar. [Afzal et al. \(2014\)](#) researched early-age autogenous shrinkage deformations in concrete containing bentonite clay by internal curing method. In the study, bentonite was used as a cement substitute at 5%, 10%, 15% and 20%, and linear shrinkage behavior was observed in concrete beams. The results demonstrated that beams prepared with 10% bentonite substitution gave the best results among all other mixtures. [Zivica et al. \(2016\)](#), determined that metabentonite-based geopolymers are a material with super strength effect.

[Adeboje et al. \(2020\)](#) examined concrete containing bentonite clay and rubber. The results revealed that the concrete containing bentonite clay-crumbled rubber showed mechanical and microstructural properties close to the concrete in the control mixtures. [Masood et al. \(2020\)](#) investigated the mechanical and durability properties of a RCA containing bentonite in concrete. Results showed that the compressive and splitting tensile strength of concrete containing 15% bentonite increased and chloride penetration decreased. [Ashraf et al. \(2022\)](#) evaluated the mechanical and durability properties of concrete containing bentonite clay and silica fume. In the study, 10% silica fume and 7.5%, 15% and 22.5% bentonite were used to replace of traditional Portland cement. With the substitution of bentonite and silica fume, the resistance of concrete to sulfate and chloride resistance has increased significantly. [Ul-Haq et al. \(2022\)](#) evaluated the mechanical and durability of samples containing bentonite, silica fume and polypropylene fibers. In the research, in addition to binary mixtures containing bentonite or silica fume as a cement substitute, triple mixtures were also acquired. In addition, different concrete mixtures were obtained by adding polypropylene fibers to triple mixtures in 0.25% increments. Results indicated that the addition of silica fume and bentonite improved mechanical properties compared to the control mixture.

In this study, the aforementioned physical and mechanical experiments were carried out on geopolymer mortars for metabentonite and slag-based recycling concrete aggregate. As mentioned, bentonite is a widely available material and at the same time low in cost. Considering the concrete or mortars produced with Portland cement, geopolymer mortars or concretes are materials with a high potential to develop sustainable materials. However, geopolymer technology is an alternative not only in terms of environmental and economic aspects but also in terms of superior mechanical and durability properties. In addition, the use of recycled aggregates in reducing the environmental impact of construction activities in our country within the scope of urban transformation projects has been increasing in recent years, and regulations regarding quality and use are included in the standards. In this context, as a result of the study on the production of geopolymer mortar by using both by-products and recycling aggregate replacement of sand, experiments were performed and satisfactory results were obtained.

MATERIALS AND METHODS

Raw materials and characteristics

Specific gravity of the GGBFS is 2.9 and the amount passing through a 45-micron sieve is 98.6%. The

information on the chemical composition of the slag is presented in Table 1. Bentonite's specific gravity is 2.42. The montmorillonite content of bentonite is at least 60% and the lime content is at most 1.2%. The calcining process of bentonite was carried out in a high-temperature furnace. During this calcination process, bentonite was brought to a temperature of 900 °C for 5 hours. Recycling concrete aggregate (RCA) obtained from the company was

passed through a 2 mm sieve and made ready for use in geopolymer composite production. The specific gravity of the waste aggregate is 2.05 and its chemical composition is given in Table 2 below. NaOH and Na₂SiO₃ were used as chemical activators. The purity value of NaOH is over 99%, the percentage of Na₂SiO₃ is 27.2 SiO₂, 8.2 Na₂O and the pH value is between 11 and 12.4.

Table 1. Chemical properties of slag.

Chemical Composition	SiO ₂	Al ₂ O ₃	Fe ₂ O ₃	TiO ₂	CaO	MgO	K ₂ O	Na ₂ O	LOI
GGBFS	40.55	12.83	1.1	0.75	35.58	5.87	0.68	0.79	0.03

GGBFS: Ground granulated blast furnace slag

Table 2. Chemical properties of recycling concrete aggregate.

Chemical Composition	SiO ₂	Al ₂ O ₃	Fe ₂ O ₃	TiO ₂	CaO	MgO	K ₂ O	Na ₂ O	MnO
RCA	62.56	12.52	5.82	0.75	12.01	1.83	1.3	2.69	0.12

RCA: Recycling concrete aggregate

Geopolymer mortar mixtures

In the study, geopolymer mortars were designed to be in 10-12M and different GGBFS: MB ratios (100 MB, 75 MB+25 GGBFS, 50 MB+50 GGBFS, 25 MB+75 GGBFS). The mixtures were prepared with a sand/binder ratio of 2.5, an activator/binder ratio of 0.85, and a Na₂SiO₃/NaOH ratio of 2:1 by weight. In this regard, both the studies in the literature and the results of the preliminary trials in the laboratory were used.

For the preparation of geopolymer mortars, alkali activators NaOH solution and sodium silicate were mixed and cooled at ambient temperature. MB, GGBFS, and RCA were mixed thoroughly for 5 minutes and the activator was added at ambient temperature. In the last stage, all mixtures were mixed at low speed for 2 minutes. After the samples poured into the molds were kept at standard room conditions for 24 hours, they were removed from the mold and kept in an oven at 80 °C for 24 hours.

Table 3. Mixing amounts of geopolymer composites (g).

	Metabentonit	Slag	RCA	NaOH	Na ₂ SiO ₃
100MB	450	0	1125	127.5	255
75MB+25GGBFS	337.5	112.5	1125	127.5	255
50MB+50GGBFS	225	225	1125	127.5	255
25MB+75GGBFS	112.5	337.5	1125	127.5	255

GGBFS: Ground granulated blast furnace slag

Test procedure

Geopolymer samples were kept in an oven for 72 hours according to ASTM C 642-21 (2013) standard and oven dry values were taken. After 72 hours of oven-dry weighing, the samples were kept in a container filled with water for 48 hours to become saturated with water. The weights of the samples that became saturated with water were recorded. The water absorption values in percent were determined by subtracting the dry weights from the saturated weights of the samples weighed and dividing by the dry weight. After these processes, the weights of the samples in water were obtained using the Archimedes principle, and the unit weight and void ratio were determined with the help of the following formulas.

Mortars produced in 50x50x50 mm dimensions were subjected to compressive strength on the 7th and 28th days following TS EN 196-1 (2005). Three separate samples for

each series were used for compressive strength and the average was taken. Three 40x40x160 mm prism specimens were used to determine the flexural strength of the specimens. The samples will be loaded from a single point according to the standard in the automatic test machine, and the tests were carried out on the 7th and 28th days, and the flexural strength was found by taking the average of the three test samples.

Ultrasonic pulse velocity was carried out by measuring the duration of the ultrasound waves applied from one surface of the sample defined in TS EN 12504-4 (2004) and ASTM C 597(2009) standards to travel the distance between the other surface. For the probes to contact the sample surface within the scope of the experiment after the gel material was applied, three readings were made for each sample and the average of the three values was taken.



Figure 1. Compressive, flexural strength and ultrasonic pulse velocity test on samples.

RESULT AND DISCUSSION

Physical Properties

Data on unit weight and water absorption rates of MB-GGBFS-based geopolymer composites at two different molar concentrations are presented in Figs. 2-4. As it is known, geopolymer, which has a denser structure, has less porosity and fewer voids.

While the lowest void ratio value was obtained at 13.24 in 75GGBFS+25MB series and 12 M, a void ratio increase of 39.82% was observed at 100 MB. However, in the 28-day water absorption, the 100 MB series with the lowest value of 4.09 indicates that the water absorption tends to decrease as the molarity increases, while at 10M this value increased by 71.40%.

Thanks to these properties, mechanical properties also show a similar behavior tendency. It is seen that there is a parallelism between the unit weight and water absorptions and the compressive and flexural strengths.

Ahmad et al. (2011) substituted 0%, 20%, 30%, 40% and 50% by weight bentonite to examine the effect of calcined and uncalcined bentonite on the properties of mortar. For calcined bentonite, this process was carried out at 500 °C and 900 °C. The results of the study showed that the water absorption values decreased for mortars containing up to 30% bentonite, and these results increased proportionally for higher bentonite replacement ratios. In the research, when the mortars were immersed in 5% Na₂SO₄ and 2% MgSO₄ solutions, the highest compressive strength was obtained from the mortars containing 30% bentonite.

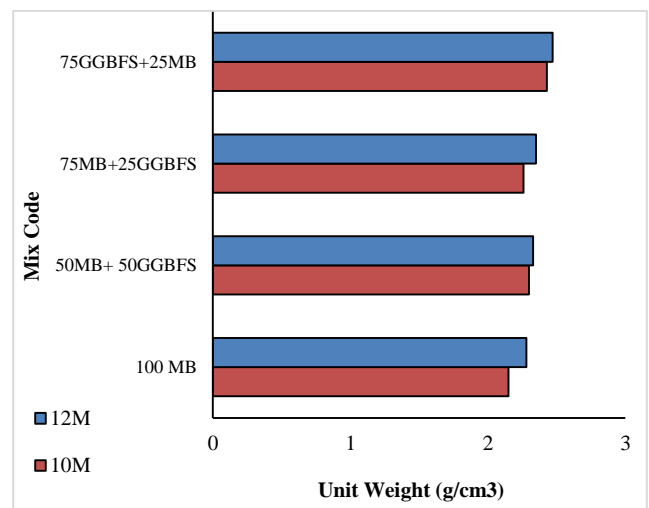


Figure 2. Unit weight results of metabentonite and slag-based geopolymer composites.

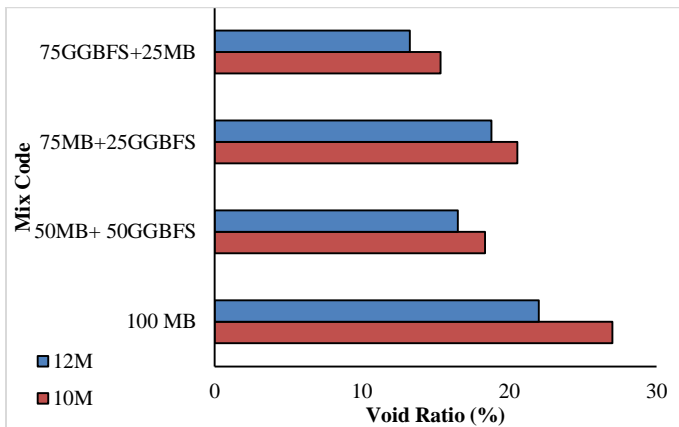


Figure 3. Void ratio results of metabentonite and slag-based geopolymer composites.

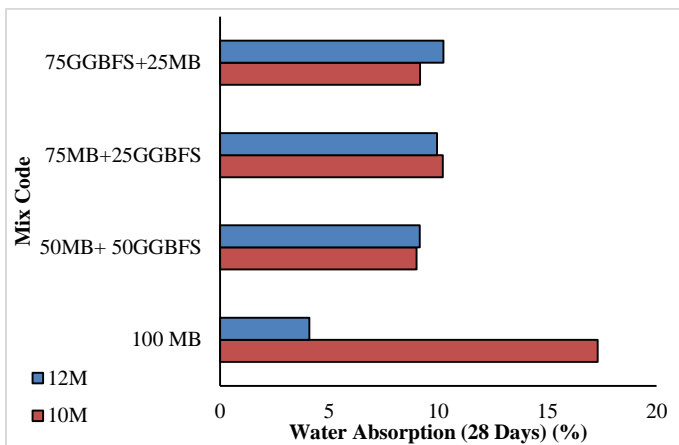


Figure 4. Water absorption results of metabentonite and slag-based geopolymer composites.

Mechanical properties

Compressive and flexural strength results of MB-GGBFS based geopolymer mortars at two different molar concentrations on the 7th and 28th days are given in Fig. 5-8. Because the raw material forms of clays and shales that show crystal structure do not show pozzolanic properties.

As it is known, bentonite used as a substitute in geopolymer shows a low pozzolanic reactivity. However, the pozzolanic reactivity increases with heat treatment at 700–1000 °C. Bentonite matrix caused a structural change in montmorillonite by applying heat treatment at 900 °C. Shape, density and structure of anhydrite montmorillonite demonstrated that the structure of meta-montmorillonite was highly preserved in crystal form. In the case of recrystallization, the formation of additional phases and an amorphous structure was achieved. The presence of

metabentonite in the study increased both strength and ultrasonic pulse velocity values due to the high Si/Al ratio.

Although the highest compressive strength values are obtained in the series with 75% GGBFS and 25% MB substituted, the difference is about 3 MPa compared to the series with 75% MB and 25% GGBFS at 10M. In the results of 28-day flexural strength, the strength difference in these mixture ratios decreased significantly. However, the difference in compressive strength increased markedly at 12M. Figures show that the mechanical properties improved with increasing molar concentration. In the 75GGBFS+25MB series, which was the highest value for 28 days in compressive strength, the closest result to 41.86 MPa was found in the 50GGBFS+50MB series, with a decrease of 7.12%. The 28-day flexural strengths showed that the difference between these two series was not obvious, and the difference did not change for the 12M and 10M concentrations. While the highest flexural strength was obtained as 10.23 Mpa in the slag-based series, the strength decreased by 2.83% in %50 replacement. The high NaOH molarity in the C-S-H phase contributed to the strength development of geopolymers by reducing the C/S ratio. The crystallization of the geopolymer was improved by the conversion of the N-A-S-H gel to crystalline hydroxy sodalite, and thus the NaOH molarity changed the degree of geopolymerization reaction. In this way, it strongly influenced the compressive strength as well as the microstructure of the geopolymers. Based on studies in the literature, it was observed that the strengths increased with the rise in GGBFS content.

In the study conducted by [Aygörmez \(2021\)](#), four different ratios of metabentonite and metazeolite were used in geopolymer mortars. Compressive and flexural strength, and ultrasonic pulse velocity tests were performed on the 7th, 28th and 90th days of the series. In addition, freezing-thawing test tests at 450 cycles and high-temperature tests on the 28th day were carried out on geopolymer mortars. With the results obtained from these tests, weight, strength and ultrasonic pulse velocities were attained. When all the results were evaluated, it was seen that the geopolymer samples prepared with metazeolite and metabentonite were resistant to the effects of strength and durability.

[Al-mashhadani and Canpolat \(2020\)](#) investigated the effect of the use of different fillers on the properties of FA-GGBFS based geopolymer composites produced at different NaOH concentrations (8M and 12M). In the study, microstructure properties such as mechanical and physical properties, abrasion and freezing-thawing

resistance, SEM and X-ray diffraction were investigated. In general, the inclusion of crushed limestone and waste foundry sand in the mixtures positively affected the overall properties of the samples. The higher NaOH concentration allowed vaguely better mechanical strengths compared to samples with lower NaOH molarity.

Chaithanya et al. [23] designed geopolymer mortar mixtures containing 80% and 20% GGBFS. Compressive strength, splitting tensile strength and durability tests were carried out by using 1:1.5, 1:2, 1:2.5 and 1:3 mixing ratios

of NaOH and Na₂SiO₃ solutions in the mixtures. According to the test results, increasing the solution concentration from 8M to 14M improved the strength properties of geopolymer concrete. Compressive strength values also increased with the rising of alkali solution concentration. When the amount of fine aggregate in the mixtures was increased, the weight loss and compressive strength loss also decreased.

Ultrasonic pulse velocity results were parallel to both compressive and flexural strengths (Figure 7.).

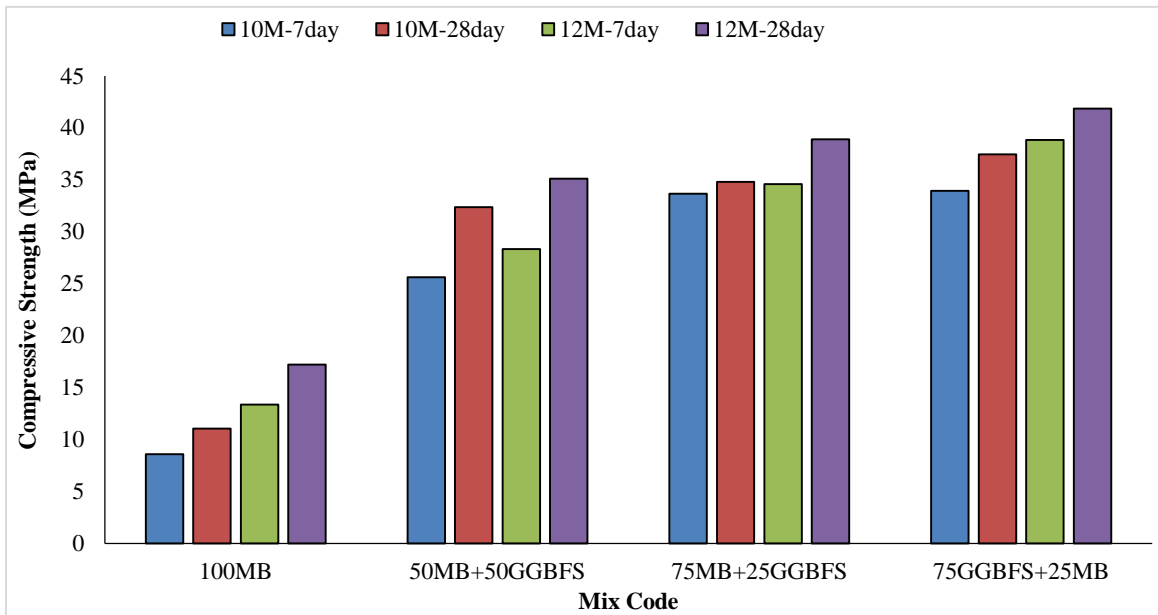


Figure 5. Compressive strength results of metabentonite and slag-based geopolymer composites.

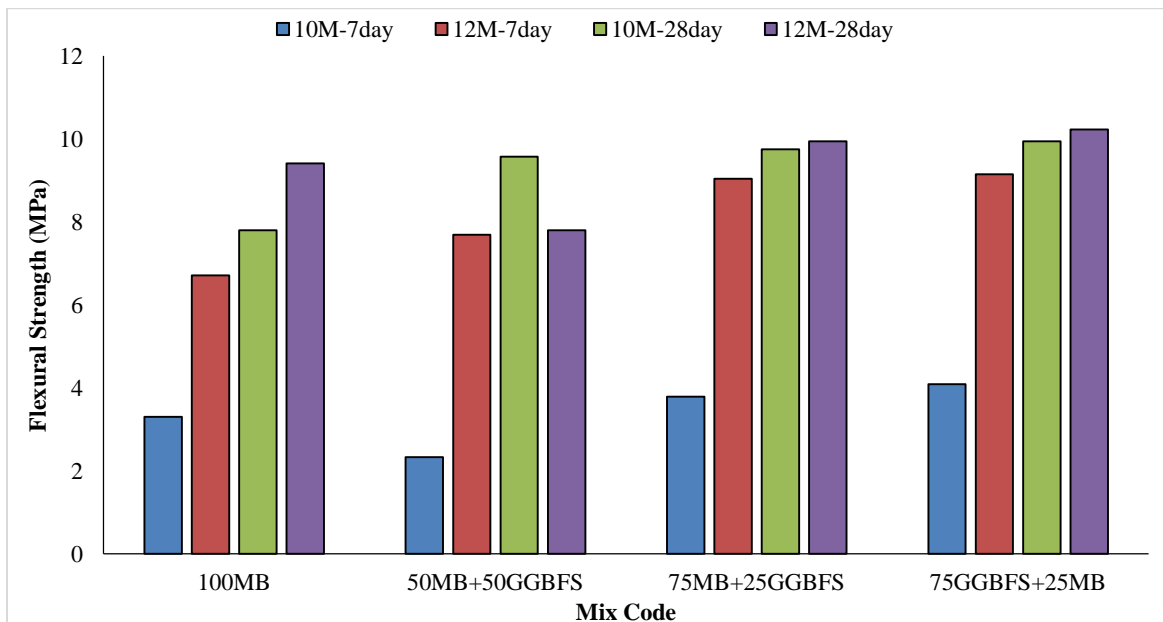


Figure 6. Flexural strength results of metabentonite and slag-based geopolymer composites.

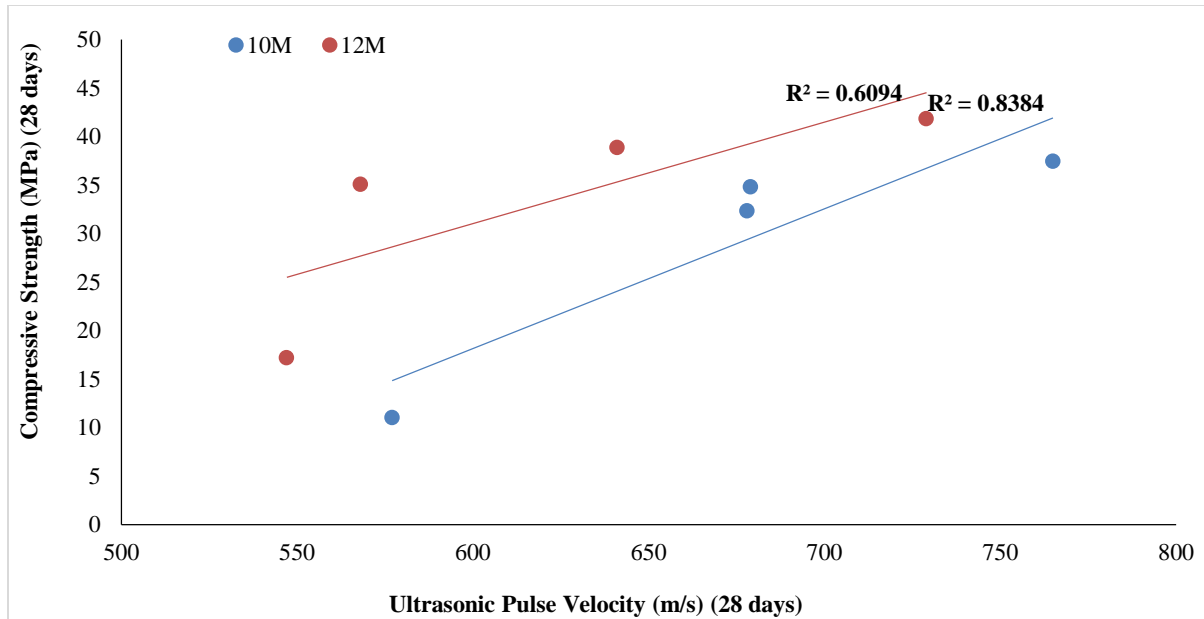


Figure 7. Relationship between compressive strength and UPV in metabentonite and slag-based geopolymer composites.

CONCLUSIONS

Considering the negativities that our world is exposed to due to global climate change, mortars and concretes with geopolymer binders must become widespread in our country and the world to minimize the damages caused by the greenhouse gas effect caused by cement production.

- By using the recycling aggregate in the production of geopolymer composite, it has been transformed into an alternative product to the standard aggregate. The rubble obtained from the buildings that collapsed or are destroyed in the earthquakes that occur frequently in our country and which are found to be risky within the scope of urban transformation must be converted into recycling aggregate and used in geopolymer technology as an economical product.

- By using “meta” bentonite, which is produced by calcining bentonite, in the production of geopolymer, positive results were obtained in the physical properties of geopolymer composites such as unit weight, water absorption and void ratio, and mechanical properties such as compressive, flexural strength and ultrasonic pulse velocity.

Calcination at 900 °C was associated with a pozzolanic feature. The presence of semi-amorphous aluminosilicate structure enhanced geopolymerization and gained serious mechanical properties. It increased geopolymerization. In this way, important mechanical qualities were acquired.

- The molarity of the NaOH solution had a significant effect on void ratio, water absorption, compressive strength and flexural strength in temperature-cured metabentonite-slag geopolymer composites.

- While the highest unit weight value was obtained as 2.43 g/cm³ in 75 GGBFS+ 25 MB series, the water absorption rate of this series was 9.16% for the 10M.

- However, it was determined that when the molar concentration increased to 12 M, especially for the 100 MB series, the void ratio and water absorption values decreased significantly.

- A remarkable structural strength was achieved in all series except the 100 MB series. While the 7-day compressive strengths of these series at 10M concentrations varied between 26-36 MPa, their 28-day compressive strengths were between 32-38 MPa.

- For flexural strengths that exhibit similar behavior to compressive strengths, flexural strengths of 7 and 28 days were in the range of 7-10 MPa, except for the 100 MB series.

- When the NaOH molar concentration increased from 10M to 12M, the compressive strength increased by approximately 15% and the flexural strength by 5% for all series.


- It was observed that there is a strong correlation between compressive and flexural strength and ultrasonic pulse velocity.

Suggestions

- Geopolymer studies can be increased in order to recycle the blast furnace slag and fly ash wastes, which can be the main components of the geopolymer material, and to observe the economic and environmental advantages.
- Different curing method can be developed considering both the application and cost of heat curing for the geopolymer to gain strength.
- Studies can be developed to recycling concrete aggregate used within the scope of the research and the wastes to be generated in the urban transformation projects of the country.
- Effect of fiber additives on the strength and durability of geopolymers can be investigated in more detail for a longer period of time. In particular, the effect of the dimensional structure of the fibers used on the strength and durability can be examined.

DECLARATIONS

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Author's contribution

All authors contributed equally to this work.

Data availability

The datasets used and/or analysed during the current study available from the corresponding author on reasonable request.

Competing interests

The authors declare no competing interests in this research and publication.

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