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Research Article

The influence of microwave curing on the strength of silica fume-added fly ash-based geopolymer mortars

Berivan YILMAZER POLAT

Department of Architecture, Faculty of Fine Arts, Design and Architecture, Munzur University, 62100, Türkiye

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ABSTRACT

Geopolymer concretes a substitute for traditional Portland cement as a more environmentally sustainable alternative. In addition, one of the most significant advantages of geopolymer concrete is the high setting speed that can be achieved with thermal curing, that is, the short curing period. Research on geopolymer concrete has focused on curing, where curing is usually done with conventional ovens. Microwave curing initiates heat generation at the molecular level, which results in a minimal heat increase on the surface of the specimens compared to traditional thermal curing methods. In addition, it provides internal, rapid, and homogeneous heating and rapid strength development. This study is based on research that examined the effect of microwave cure on the compressive strength of silica füme-modified geopolymer mortar. In the study, sodium silicate and sodium hydroxide were used for activation, and first of all, fly ash substituted geopolymer mortar specimens were subjected to conventional thermal curing in an oven. On the other hand, the mortar specimens with the same composition were subjected to short-duration (10 minutes) microwave curing at power levels of between 200-600 W. When the results were compared regarding compressive strength, void and percent water absorption, UPV, and SEM-internal structure examination, it was revealed that microwave curing could be used faster than the conventional oven curing method in silica füme-added fly ash-based geopolymer mortars. This research can be considered an essential step for further optimizing and popularising geopolymer concrete and microwave curing and proposes a method that should be considered in the future development of more sustainable construction materials.

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1. INTRODUCTION

Concrete has long been a fundamental building material extensively utilized in the construction and building sectors. Traditionally, concrete is formed by blending aggregates, water, and Portland cement through a process known as hydration. However, the production of Portland cement is associated with a significant release of CO_2 into the environment, posing environmental hazards and sustainability concerns. It's estimated that every 1 kg of cement production emits 0.87 kg CO_2 [1]. Consequently, the quest for alternative binding agents has become crucial, attracting

***Corresponding author.**
*E-mail address: berivan_ist@hotmail.com

the attention of numerous researchers. At this juncture, geopolymer mortars have emerged as a solution, reducing carbon emissions by up to eighty percent.

Additionally, they lower the energy consumption related to cement production by around thirty percent [2]. Another advantage is its contribution to sustainability. Geopolymer concretes can be produced by activating many waste materials such as fly ash (FA), metakaolin, blast furnace slag, silica füme (SF), and rice husk ash [2–5]. Moreover, the most frequently used ones are FA, metakaolin, and slag. FA and slag SF, which can be used in geopolymer mortars, are recyclable materials obtained as by-products.

Fly ash, a fine powder derived from burned coal or coal slag typically forms during thermal power generation and serves as a primary binder component in geopolymer concrete production. Using fly ash in geopolymer concrete helps two primary purposes: it aligns with sustainability goals, providing an advantage to geopolymer concretes. Secondly, it exhibits activatable binder properties. By repurposing burnt coal or coal slag—otherwise considered waste—fly ash's incorporation in geopolymer concrete production aids in conserving natural resources and mitigating environmental impacts. Simultaneously, as a binding material in geopolymer concrete, fly ash actively solidifies through hydraulic reactions, resulting in a durable structure. The aluminosilicate compounds within fly ash interact with alkali activators, generating a polymeric structure that fortifies the concrete. Consequently, geopolymer concrete boasts elevated compressive strength, improved chemical resistance, and reduced permeability.

FA standards (ASTM C 618 and TS EN 197-1) divide FA material into two groups. ASTM C 618 defines pozzolanic ash with a CaO percentage below 10% (low calcareous and $SiO_2+Al_2O_3+Fe_2O_3>70\%$) for class F FA, while the percentage of CaO for class C FA is more than 10% (high calcareous). And $SiO_2+Al_2O_3+Fe_2O_3$ >50%). Class C FAs are pozzolanic and binder [6]. Conversely, TSE divides FAs into siliceous (V) and calcareous (W) groups. While V class FA is defined as reactive lime and active silica (CaO<10%, SiO2>25%), W class FA is defined as containing active limestone and reactive silica (CaO>10%, SiO2<25%) [7].

Like FA, SF can be used at different rates in geopolymer mortars. It has advantages such as improving the mortar's mechanical properties, reducing its porosity, improving its properties, and contributing to thermal comfort, depending on the purpose. Silica fume is a fine powder in which silica or silicon dioxide (SiO_2) particles are suspended in the air. It is usually formed by the oxidation of silicon or silicon compounds at high temperatures. Silica fume consists of very fine particles; this fine particle structure provides a homogeneous material distribution. High surface area increases chemical reactivity and improves adhesion. This increases the adherence, making the concrete more durable and cracking-resistant. In addition, since it is generally a chemically inert material, it contributes to the chemical stability of conventional concrete at high temperatures compared to geopolymer concrete. The SF used in this study was added to the mixture due to this thermal stability feature. Microwave curing tends to increase in temperature at high powers, causing the specimen to crack. Adding SF to the mortar is aimed at partially preventing these microcracks. In addition to all these material properties, the use of FA and SF in this study supports research to help reduce the amount of environmental waste and increase environmental sustainability.

The most frequently used activators are sodium hydroxide, potassium hydroxide, and sodium silicate.

The reaction takes place in the following steps in FA-added geopolymer mortars.

- Use of activators to activate Al and Si atoms
- Combination of solid FA particles with Al and Si atoms to form aluminosilicate gel
- Polymerization formation
- Condensation

Calcium silicate hydrate gels are also found in conventional Portland cement products. For example, the most common activators in the gels that provide the binder hydration process are sodium hydroxide, potassium hydroxide, and sodium silicate [8].

The reaction and geopolymerization process is directly affected by the curing temperature and activator concentration [9]. Polymerization takes place very slowly in geopolymer mortars that are left to cure with their heat of hydration. For this reason, in most cases, the curing environment is heated by an external heat source without losing the moisture of the mortar. However, these methods generally require 1-7 days of curing. On the other hand, microwave ovens use electromagnetic microwave energy to emit vibrations from the inside out at the molecular level and provide heating very quickly. This makes them much more suitable for prefabrication. The general working process of microwave ovens starts with generating microwave energy by a microwave generator inside the oven. Next, an unloader (magnetron) tube inside the oven receives the generated microwave energy. The magnetron tube converts electrical energy into microwave energy and spreads this energy inside the oven, creating a microwave field. The microwave field focuses on the food or material and creates a vibration at the molecular level. This molecular vibration causes the water molecules in food or materials to accelerate and heat up. The heated water molecules also heat the other molecules in their surroundings, heating the material or food [10–12].

Among the thermal curing methods used in the literature, there are studies of oven curing, hot water curing, autoclave curing, steam curing, curing methods, and a few microwave curing methods [5, 13, 14]. However, there needs to be a more detailed investigation in the literature regarding the strength aspects of FA-based geopolymers, and no research has been found that specifically explores the reaction of SF-incorporated and FA-based geopolymers with microwave curing. In this sense, this study will likely

fill an essential gap in the literature and make a strong contribution. For this purpose, FA-based geopolymer mortars containing SF additive were prepared in the study. It was determined that the effects of conventional oven curing and microwave curing on compressive strength were revealed, and optimum microwave power was determined.

2. EXPERIMENTAL WORK

Geopolymer mortars represent resilient and ecologically sound construction components that offer a viable substitute for conventional cementitious materials. These matrices are engendered through the amalgamation of fly ash, commonly augmented by alkali activators sourced from industrial residuals. In the context of this investigation, fly ash (FA) and silica fume (SF) were harnessed as principal binders. The activation process involves the introduction of sodium silicate and sodium hydroxide, serving as pivotal alkali activators, while the geopolymer mortars are constituted utilizing RILEM sand. The Class F variant of fly ash employed herein is a derivative stemming from the operational endeavors of Cenal Elektrik Üretim A.Ş. The chemical attributes of the fly ash are enumerated in Table 1 for reference and scrutiny.

Antalya Eti Krom furnished the SF. The chemical characteristics of the SF are shown in Table 2.

Other materials used in mortar are CEN-Standard RILEM Sand (EN 196-1) as aggregate. Table 3 shows the granulometry of the sand. NaOH (%99 purity, prepared in 12 moles by water dissolving with water) and Na_2SiO_3 (ρ:1,35 g/cm^3 at 20°C) are the activators. The specimens were prepared using fly ash+silica fume=1 part (90% FA+10%SF), 3 parts sand, and 0.5 part activator ratio. After allowing a 12 M NaOH solution made with water to calm fully, NaOH and Na_2SiO_3 were combined at the room's temperature in a 1:2 ratio.

First, mortar specimens were prepared for conventional and microwave oven curing. For this purpose, sand and pozzolans put into the mixer were mixed for 1 minute, then activators were added and mixed for two more minutes. It was placed in 50x50x50 mm molds for compressive strength

Table 3. The granulometry of the sand

Sieve Square Mesh (mm)	Cumulative Remainder (%)
2	0
1,6	7
1	30
0,5	67
0,16	87
0,08	99

Table 1. Chemical analysis of the FA

Chemical Components	SiO_2 (%)	Al_2O_3 (%)	MgO (%)	SO_3 (%)	Fe_2O_3 (%)	CaO (%)	Na_2O (%)	K_2O (%)	Total Alkali	Specific Gravity	Activity Index 28 days (%)
Analysis Results	59.37	21.40	2.108	0.202	8.620	3.237	1.267	1.804	2.45	2.38	78.8

Table 2. Chemical properties of the SF

Chemical Components	C_2O3	SiO_2	Fe_2O_3	Al_2O_3	CaO	MgO
Analysis Results	%3,5-5	%70-80	%1,17-5,0	%2,55-4,10	%1,06-1,80	%8,05-9,9

Figure 1. Images from the preparation stages of the specimens.

Figure 2. The conventional oven.

Figure 3. The microwave oven.

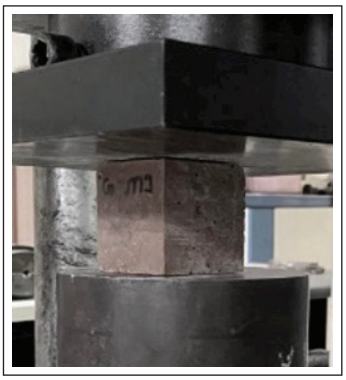


Figure 4. The compression testing machines.

testing (Fig. 1). It was kept in the mold for 24 hours at room temperature and wrapped airtightly. Afterward, some of the specimens removed from the mold were cured in a conventional oven at 60°C for 30 minutes, 60 minutes, 90 minutes, 24 hours, and 48 hours. Other specimens that needed to be cured in a microwave oven were kept for 10 minutes at 200 W, 300 W, 400 W, 500 W, and 600 W (Fig 2 and 3).

All cured geopolymer mortars were kept at room temperature without losing their moisture until the 7th day. They were subjected to a compressive strength test on the seventh day (Fig. 4). After this stage, all specimens subjected to microwave curing were compared with those cured in a conventional oven for 24 hours regarding void ratio, water absorption percentage, and UPV transmission rate.

Only the 24-hour curing comparison is considered adequate after this section, as many conventional oven-curing studies have been conducted in the literature. The experiments were performed according to TS-EN 12504-4 and TS EN 1015 -11 standards, respectively. These experiments aim to see the changes in the specimens' void ratios and water absorption values after microwave curing at different powers. Then, the samples exposed to conventional and microwave curing were examined by SEM images.

Compressive strength tests of 50 mm cube geopolymer mortars were performed at a load rate of 0.5 N/mm^2/second and on the 7th day according to TS EN 12390-3 [21] standard. Three specimens were tested for each, and mean values were used to indicate the results. The standard deviation between specimens was 2.5% (Fig. 4)

3. RESULTS AND DISCUSSION

In this study, which aims to investigate the strengthening of geopolymer mortars with the help of microwave ovens, the prepared mortars were first cured in the microwave oven at different Powers, and the temperatures of the hardened mortar specimens were measured. These temperatures are significant for the interpretation of the results. Then, these geopolymer mortars, cured in conventional ovens at different times and different powers in the microwave oven, were compared in terms of their compressive strength. Afterward, the analysis of the internal structure of the mortars after hydration was made by SEM internal structure imaging method, and all the results are given below.

As a result of the measurements made with an infrared thermometer, the temperatures of the specimens rise above 200 degrees after 10 minutes of microwave curing. Since temperatures above this degree are thought to damage the mortar's microstructure, the microwave oven's cure time is limited to 10 minutes. This effect is confirmed by compressive strength results (Fig. 5).

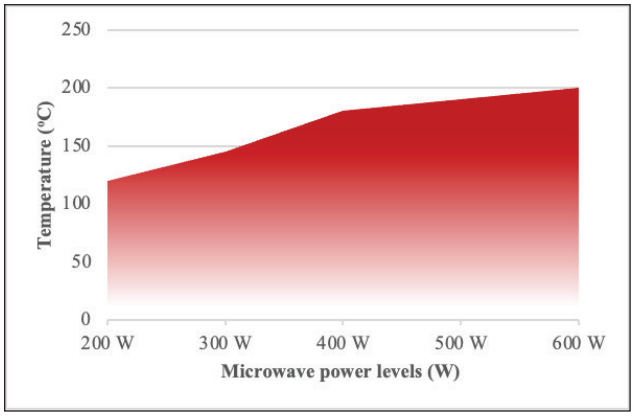


Figure 5. Final temperatures of the specimens after 10 minutes of microwave cure.

Fig. 6 shows the compressive strength test results of the specimens cured at 60°C in conventional ovens for 30 min, 60 min, 90 min, 24 h, and 48 h. According to these results, the strength only reached 15.8 MPa after 30 minutes of oven curing, while it reached 73.1 MPa after 48 hours of thermal curing. The regression between the curing time and compressive strength showed a positive increase. This situation has been demonstrated in many studies as the positive contribution of kiln curing to the hydration process in geopolymer mortars [3, 15].

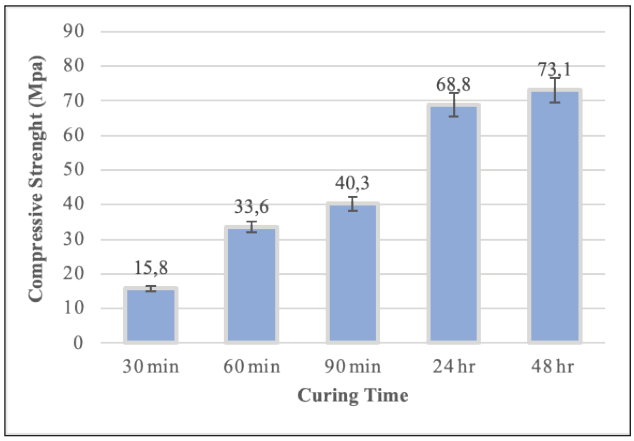


Figure 6. Compressive strength-curing time graph of the specimens after 60°C oven cure.

However, this effect corresponds to a limited point. Even in 90-degree oven curing, the maximum strength could slightly exceed 80 MPa, and the final strength ended in this range for any curing method [1, 16–18]. This situation revealed that the values obtained at the end of the traditional thermal curing given here could be affected by a maximum of 40%, independent of the content and curing temperature, but depending on the curing time variable [19, 20].

Graytee et al. (2018) found the conventional curing strength for 24 hours to 52 MPa without adding silica fume, which was measured as 68.8 MPa in this study (Fig. 7). This difference is due to the substitution of silica fume. The 48-hour strengths in both studies are closer to each other.

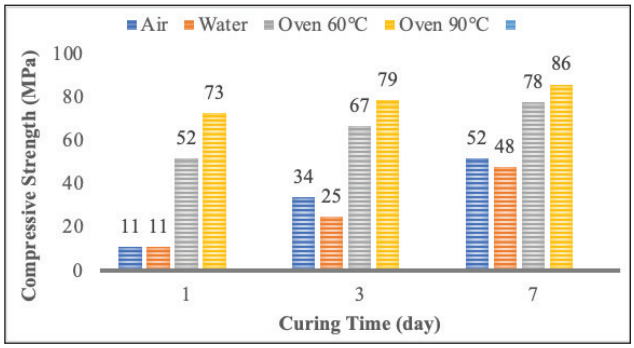


Figure 7. Compressive strength of fly ash-based geopolymers treated with different cures [18].

The compressive strength values given in Fig. 8 and measured after 200W, 300W, 400W, 500W, and 600W microwave curing did not distort this general view. The specimens cured in a microwave oven at 200 W for 10 minutes reached a strength of 39.5 MPa, while the other values were 67.7 MPa, 58.7 MPa, 50.2 MPa, and 44.8 MPa, respectively. The specimens with the highest compressive strength were cured at 300 W power. This value is approximately 8% below the strength created by the 48-hour thermal curing value, which shows the highest strength in conventional thermal curing.

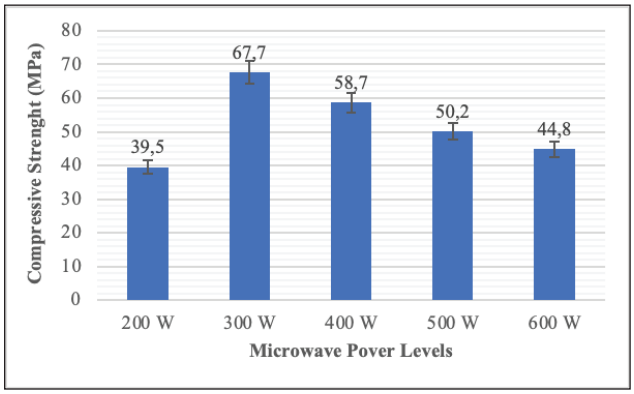


Figure 8. Compressive strengths after microwave cure of the specimens.

In another study where microwave curing was applied for 15 minutes on a geopolymer mortar specimen containing only fly ash, strengths of 59, 72, 67, and 54 MPa were obtained at 200, 300, 400, and 600 W, respectively. [18]. These strength differences have shown us that it is important to do the curing in sufficient time and at the optimum temperature and that it can change the initial and final strengths. Still, it has been observed that the curing time is similar between the specimens at maximum strength. When the two studies are compared, it is thought that the difference between the compressive strengths due to curing times is 5-10% at most. Since no research in the literature includes mixing ratios that match precisely with this study, it is necessary to conduct more comprehensive research to obtain more detailed information on this subject.

According to the experimental data of the study, curing at 400, 500, and 600 W power values above 300 W resulted in reductions in compressive strength exceeding 10%. El-Feky et al. (2020) reported that a 25-30 MPa compressive strength occurred in microwave-curing slag-based geopolymer specimens performed for 6 minutes with a power value of 720 W [21]. This result shows that high-power curing damages the structure of geopolymer mortar, consistent with our study. In addition, in cases where the curing exceeds 300 W, the increase in temperature to values close to 150 degrees causes the moisture loss of the specimens to be high, which leads to the formation of cracks in the specimens (Fig. 9). Crack formation is possible due to rapid moisture loss at high temperatures. In cases where curing is done much faster than the conventional method, such as microwave curing, it may pose serious risks and affect the concrete strength [22]. When this risk is controlled, microwave curing can provide an alternative to the traditional method for silica fume-added fly ash-based geopolymer mortars.

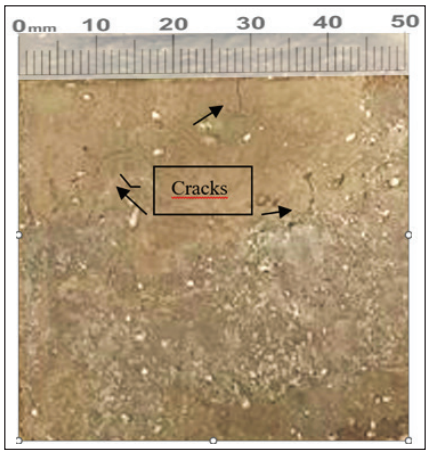


Figure 9. Crack image of specimen cured at 600 W.

Considering the compressive strength test results of curing the specimens with microwaves at different powers, Fig. 7 and 9 show that the activation of fly ash-based silica fume-added geopolymer mortars could develop faster and better strength with microwave oven curing without heat cure.

The measured void percentages, water absorption percentages, and UPV values of the specimens are shown in Figure 10. The void ratios vary between 14.33% and 20.12%, the water absorption ratios vary between 6.90% and 11.47%, and the UPV values range between 2388 and 2734. As seen in Figure 10, while the highest UPV value is in 300-watt microwave curing, this curing condition also shows a close correlation with the lowest void and water absorption values (Figure 11). This value also gave the highest value in terms of compressive strength. The second best-performing specimen was 24-hour oven curing for all four parameters. The compressive strength-UPV correlation graph of the specimens also confirmed a relationship between the void amount and strength of the specimens, together with the high correlation value in this sense. As it is known, as the UPV value increases, the material's void ratio increases in the compressive strength [23–25].

The specimens that gave the worst results in these tests were those cured at 200-watt power and those cured at 600-watt power. According to this result, 200-watt power is insufficient for hydration, and 600-watt power overheats the specimen, causing cracks and disrupting stable calcite structures. SEM images of critical specimens were taken to support this interpretation.

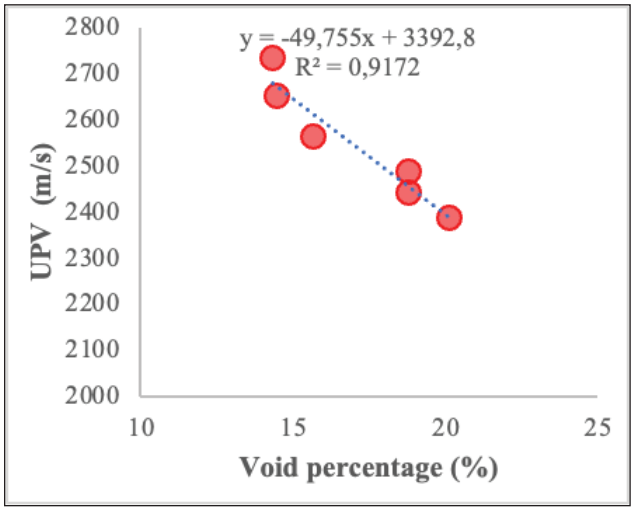
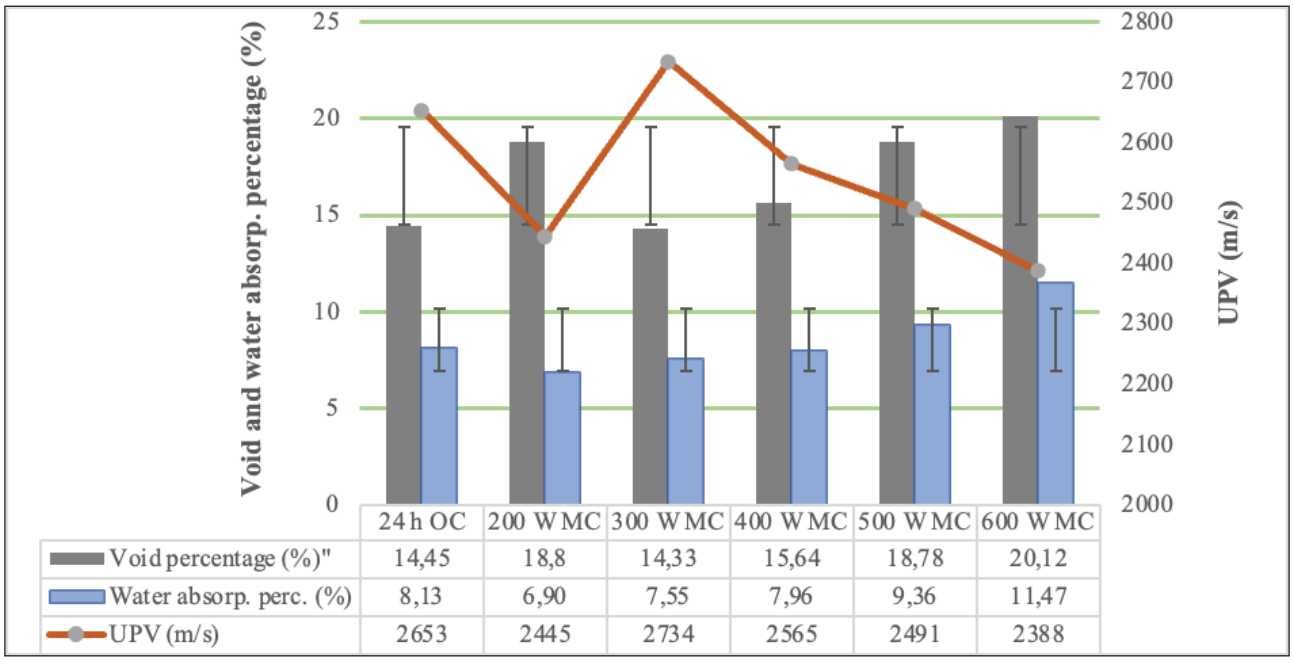


Figure 11. Correlation between void percentage and UPV of specimens.



	24 h OC	200 W MC	300 W MC	400 W MC	500 W MC	600 W MC
Void percentage (%)"	14,45	18,8	14,33	15,64	18,78	20,12
Water absorp. perc. (%)	8,13	6,90	7,55	7,96	9,36	11,47
UPV (m/s)	2653	2445	2734	2565	2491	2388

Figure 10. Void, water absorption, and UVP pass rates of the specimens.

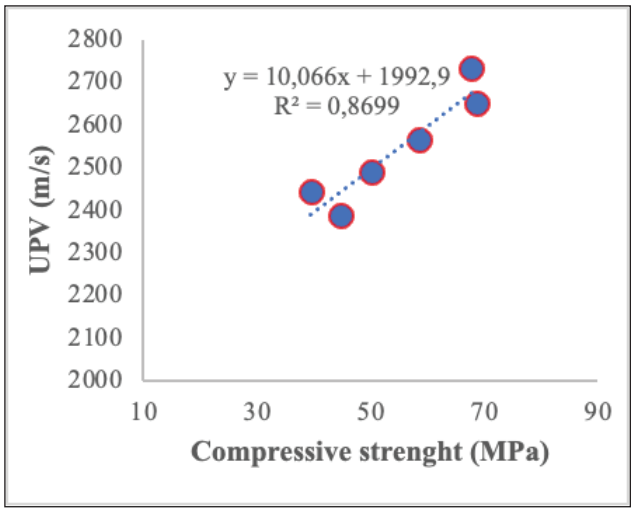


Figure 12. Correlation between compressive strength and UPV of specimens.

SEM micrographs of the specimens are shown in Fig. 13, 14, 15, and 16. SEM analysis was performed after the compressive strength tests to examine the microstructural changes of the geopolymer specimens.

In the control specimen, whose internal structure is shown in Fig. 13, gel formation on fly ash particles and hardened structure with micro gaps filled with silica fume have indicated the breakdown of the glassy phase in the alkali solution. Additionally, partly reacted fly ash particles were also seen.

As shown in Fig. 14, in the specimens with the highest strength cured 300 W, there are $CaCO_3$ structures that have hardened due to microwave radiation from fly ash and accelerated dissolution of Si and Al species. This explains the increase in compressive strength.

Figures 15 and 16 show that fissures, dissolved structures, and a constant accumulation of geopolymer products filled fly ash voids were detected. Moreover, rapid curing and evaporation caused cracks to appear.

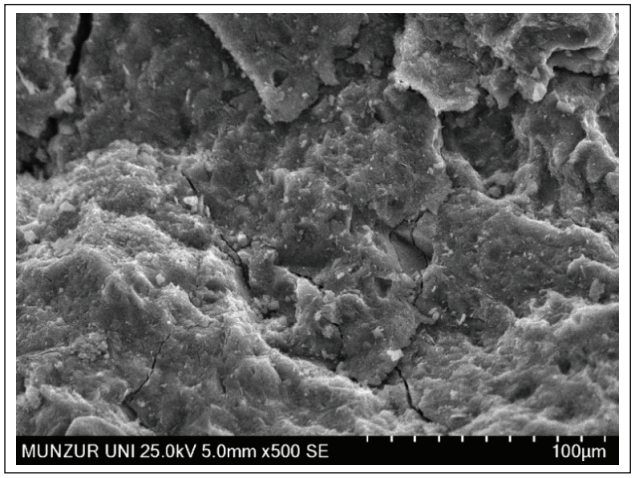


Figure 13. 60°C oven cure.

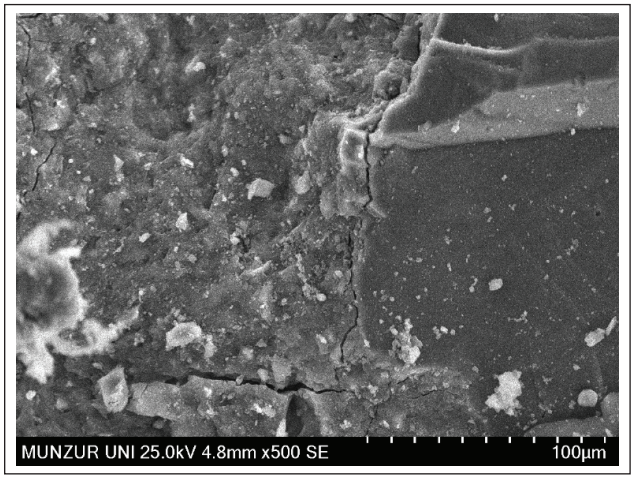


Figure 14. Microwave cure-200 W.

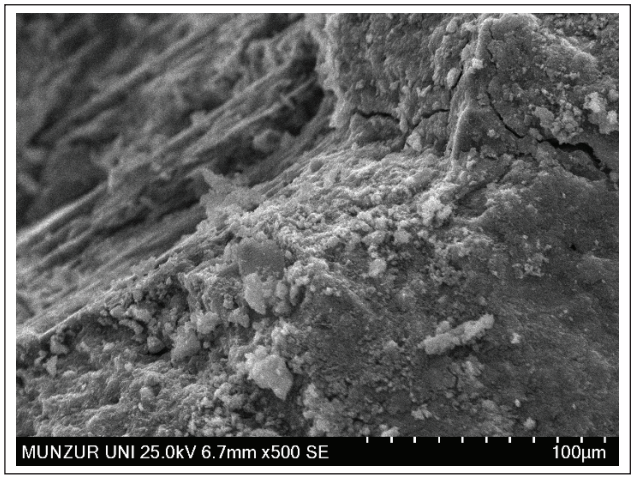


Figure 15. Microwave cure-400 W.

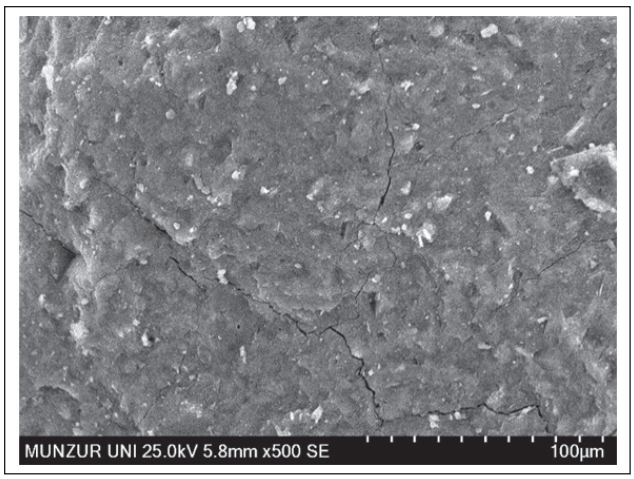


Figure 16. Microwave cure-600 W.

The following might describe the mechanism of microwave radiation's contribution to geopolymer cure. Microwave radiation affects the strength development of geopolymer before conventional heat curing. The geopolymer gel was made in part because of early microwave radiation. Because the aqueous solution heated up fast, using the microwave on specimens increased the solubility for Si and Al species from fly ash.

4. CONCLUSIONS

This research investigated the strength and internal structure properties of silica fume additive fly ash-based alkali active mortars after curing with microwave radiation. As a result, the following findings were revealed:

- It is a fast and convenient curing regime for fly ash-based geopolymer mortars reinforced with silica fume in microwave curing. In addition, 200 W power and 10 minutes of curing time are the optimum rates for this research. When these mortars are cured in the microwave, they reach high early strength values of 67.7 MPa. At the same time, this form of curing improves the microstructure. However, strength loss begins due to a high-temperature increase in high-power curing over 300 W. As it rises to 600 Watts, the mortar loses approximately 20% of its maximum strength.
- It was understood from the study results that silica fume increases the compressive strength of geopolymer mortar. Still, more detailed internal structure examination and experimental studies at different curing times are required to learn the reasons for the real contribution of silica fume on microwave curing.

The traditional curing method complicates the production process of geopolymer concrete, which is very suitable for prefabrication production. The 1- or 2-day periods spent in the furnaces for curing necessitate creating many large furnaces. This limits mass production. However, this time can be reduced to 10 minutes in curing with the microwave method. This provides a faster, less costly, and energy-efficient process for producing geopolymer mortars. In addition, this concrete, produced with waste materials such as silica fume and fly ash, can take its place in sustainable building materials and be widely used as a more environmentally friendly concrete with less energy.

In future studies, the effects of microwave curing on the internal structure and durability of fly ash-based mortars or all other geopolymer mortars should be investigated in detail. Thus, a more detailed discussion can be carried out on the subject before production.

ETHICS

There are no ethical issues with the publication of this manuscript.

CONFLICT OF INTEREST

The author declared no potential conflicts of interest with respect to the research, authorship, and/or publication of this article.

DATA AVAILABILITY STATEMENT

All graphs and data obtained or generated during the investigation appear in the published article.

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There was no specific grant for this research from the public

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