

Microstructural investigation of geopolymer composites obtained from recyclable waste materials

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Abstract: Today, researchers are exploring materials that could replace cement and provide solutions for utilizing waste products. In this study, a binder was formulated using 85% blast furnace slag and 15% fly ash. The marble powder and strontium as mineral wastes were incorporated as partial replacements to blast furnace slag at ratios of 5% to create geopolymer mortars. Also, titanium dioxide was added at a rate of 1%. The influence of these additives on the flexural and compressive strength of the mortars was assessed. In addition, SEM/EDX analysis was performed to examine the microstructural effects of the additives. The results showed that titanium dioxide achieved the highest compressive strength, recorded at 65.7 MPa. The SEM/EDX analysis further indicated that the samples displayed a homogeneous internal structure, suggesting a strong bond among the components within the matrix.

Keywords: Geopolymer; Blast furnace slag; Fly ash; Strontium; SEM/EDX

1. Introduction

The development and use of low-carbon or carbon-free products, as well as waste recycling, are particularly important in the construction industry. [1]. Given the environmental impacts, high production costs, and substantial energy consumption associated with Portland cement, the importance of alternative binders has become even more pronounced. In the current landscape of skyrocketing fuel costs, and the limited availability of fossil fuel resources (such as coal and natural gas), the construction industry has grown increasingly reliant on these sectors. While Portland cement remains the traditional and widely utilized binder in construction, its usage has come under scrutiny over the past decade, particularly due to its environmental ramifications. Recently, the production and application of binders known as geopolymer concrete and mortar have gained prominence [2]. Geopolymer applications have attracted interest for various reasons, including cost reductions through the use of waste materials during production, a contribution to lower CO₂ emissions, and performance characteristics comparable to those of Portland cement. However, it is essential that geopolymer binders used in this field fulfill the necessary performance expectations regarding mechanical properties, along with their

environmental benefits. Consequently, researchers have shown a keen interest in examining the chemical and physical properties of geopolymer concretes produced through various methods, alongside conducting strength and durability tests [3].

Naskar and Chakraborty [4] developed geopolymer concrete using fly ash with low calcium content, due to the negative environmental impacts associated with traditional Portland cement. In their study, they incorporated mixtures containing 0.75%, 3%, and 6% nano-silica, 0.02% carbon nanotubes, and 1% titanium dioxide as substitutes for fly ash in the concrete production process. The results of the compressive strength tests indicated that the additions of nano-silica and carbon nanotubes did not significantly enhance the compressive strength at 7 and 28 days. However, the inclusion of titanium dioxide at a concentration of 1% improved the compressive strength by up to 33% at 7 days, and 47% at 28 days. In a study by Nergis et al. [5], a coal ash-based geopolymer was synthesized using mine wastes activated with phosphoric acid. Three types of aluminosilicate sources were employed either as standalone raw materials or combined to create five different types of geopolymers activated with H₃PO₄ in a 1:1 weight ratio. The thermal behavior of the geopoly-

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mers activated with H_3PO_4 was found to resemble that of those activated with a mixture of NaOH and Na_2SiO_3 within the temperature range of 20-300 °C. Furthermore, within the temperature range of 400-600 °C, the geopolymers containing mining waste exhibited exothermic reactions, while those without the addition of mining waste did not show significant phase changes. In a study by Chithambaram and Kumar [6], the impact of granulated blast furnace slag (GBFS) on the partial replacement of fly ash in geopolymer mortar production was examined. The geopolymer mortars were created using a combination of sodium hydroxide (NaOH) and sodium silicate as the alkaline activator solutions. The production process involved varying the concentrations of NaOH at 8M, 10M, 12M, and 14M, along with replacing fly ash with GBFS at different percentages: 0%, 10%, 20%, 30%, and 40%. Additionally, the study established thermal curing intervals ranging from 600°C to 1000°C for the geopolymer mortar, which utilized 100% fly ash as the sole binder. Upon reviewing the results of the geopolymer mortar made with 100% fly ash, cured at 90°C using a 12M NaOH concentration, researchers found a maximum compressive strength of 51.55 N/mm². In a study where fly ash was added to ground blast furnace slag, high calcium fly ash with a particle size of 21.26 µm, blends containing fly ash in proportions were compared with samples containing 100% ground blast furnace slag. It was reported that workability performances such as plastic viscosity, segregation resistance, and flowability increased especially in mixtures containing 40%, 50%, and 60% fly ash [7].

A group of researchers [8] explored the utilization of fly ash (FA) and granulated blast furnace slag (GBFS), both aluminosilicate by-products, in the development of geopolymer cement. They conducted a comparison of six geopolymer cement mortars with varying ratios of FA to GBFS against ordinary portland cement (OPC), and magnesium potassium phosphate cement (MPPC) mortars. The study evaluated several aspects, including workability, setting time, strength development, volume stability, and chloride permeability. The results revealed that geopolymer mortars containing a high proportion of GBFS demonstrated rapid setting and significant early strength, though they exhibited some issues with volume stability. Given these findings, it was suggested that GBFS-blended mortars could serve as a more cost-effective alternative to the higher-priced MPPC mortar. In the study conducted by Hager et al. [9], the researchers examined the impact of high temperatures on the mechanical properties and microstructure of geopolymer mortars. The investigation included blends of fly ash with blast furnace slag at four different ratios. The results showed that the mixtures without slag exhibited superior performance, particularly at elevated temperatures. Flexural strengths increased up to 200 °C but demonstrated a nearly linear decline from that point to 600 °C. This decrease was attributed to dehydration processes that occur as the temperature rises. Such dehydration results in thermal shrinkage around the aggregates, which leads to cracking. At 573 °C, the

expansion of quartz sand and the transformation between β -quartz and α -quartz formations resulted in material deformation. Notably, an increase in compressive strengths was observed after reaching 800 °C.

In a study conducted by Başpınar and Kurtuluş [10], fly ash was utilized as the primary material for a geopolymer. Hydrogen peroxide was employed as a foaming agent. Various sample series were prepared by varying the mixing ratios of fly ash and blast furnace slag. The physical and mechanical properties of these samples were tested, mineralogical and microstructural characterizations were conducted using X-ray diffraction (XRD) and scanning electron microscopy (SEM) techniques. It was found that an increased addition of blast furnace slag resulted in crack formation. However, incorporating coarse aggregate significantly reduced drying shrinkage, and helped prevent crack formation. Furthermore, it was observed that adding coarse aggregate contributed to a decrease in the density of the geopolymer foam concrete blocks. The conversion of industrial by-products into geopolymer concrete is crucial for developing environmentally sustainable structural components. In a study conducted by Raj et al. [11], dolomite and ground granulated blast furnace slag were utilized as aluminosilicate materials, while NaOH and Na_2SiO_3 solutions served as alkali activators. The study highlighted that numerous parameters influence the production of geopolymer concrete, necessitating extensive experimentation to identify the optimal sample. To address this, the Taguchi statistical method was employed to determine the effective parameters and optimal samples in geopolymer concrete production. Various parameters, such as mixture ratio, molarity, and activator ratio, were compared within the L9 test series. Upon examining results related to workability, compressive strength, and splitting tensile strength of the geopolymer samples, it was found that the binder ratio was the most significant factor. Additionally, SEM microstructure analyses revealed that these structures were more compact. In geopolymer concrete, the use of source materials with low calcium content, such as metakaolin and class F fly ash, results in the formation of sodium aluminosilicate hydrate (N-A-S-H) products. Conversely, when materials with high calcium content, like blast furnace slag and class C-fly ash, are utilized, the reaction produces calcium aluminosilicate hydrate (C-A-S-H) gel products [12]. In a study conducted by Chokkalingam et al. [13], waste ceramic powder and granulated blast furnace slag were used as geopolymer binders to examine the effects of various parameters, including binder content, ratio of main binders, ratios of alkali activator solutions, and molarity. The findings revealed that using ceramic powder as a binder on its own had a minimal impact, but when combined with slag, there were significant improvements in performance. The researchers developed an L16 orthogonal array that included five factors and four levels. The analysis of variance (ANOVA) results highlighted that the ratio of alkaline activator to binder and the percentage of slag were the most influential factors affecting the compres-

sive strengths after 28 days. In contrast, the binder content and molarity had the least effect. Microstructure analyses indicated nearly equal ratios of Ca/Si and Si/Al, suggesting the presence of similar calcium hydrates and silicate phases. This was believed to contribute to the formation of calcium silicate hydrate (C-S-H) and calcium aluminosilicate hydrate (C-A-S-H) gels, which enhance the microstructure of the geopolymer concrete. It was noted that a higher Na/Si ratio compared to Ca/Si in the structure may lead to the predominance of N-A-S-H gel over C-A-S-H gels, a characteristic influenced by the presence of marble dust.

In recent years, many studies have focused on the development of geopolymer composites using industrial by-products such as fly ash and blast furnace slag. However, the incorporation of regionally sourced, underutilized mineral wastes into geopolymer systems remains limited. This study focused on incorporating marble powder and strontium-bearing mineral waste from the Sivas region of Türkiye into geopolymer mortar. The binder for the mortar is composed of 85% blast furnace slag and 15% fly ash. Moreover, titanium dioxide was utilized as an additive. The simultaneous evaluation of these three additives under identical curing and testing conditions represents a novel comparative approach in the literature. Furthermore, the inclusion of long-term curing results (up to 90 days), supported by microstructural analysis via SEM/EDX, offers valuable insight into the time-dependent behavior of these composites. Notably, the finding that a low dosage (1%) of TiO_2 can significantly enhance early compressive strength (up to 65.7 MPa) contributes to the optimization of performance with minimal additive use. The study thus fills a crucial gap by proposing a sustainable, high-performance geopolymer system utilizing local industrial wastes with potential for practical application

in earthquake-prone regions.

2. Materials and Methods

In the study, geopolymer mortars were created using 85% blast furnace slag and 15% fly ash (85BFS15FA), activated with a 10M NaOH solution at 75°C. Three different additives were incorporated into the mortar mixtures by replacing portions of the blast furnace slag. Due to the exothermic reaction of sodium hydroxide when it comes into contact with water, the solution was prepared one day in advance in glass containers to ensure it was ready for use. In addition to sodium hydroxide, sodium silicate was used as another alkaline activator. The two were combined to form a ready solution, with 70 grams of sodium hydroxide and 180 grams of sodium silicate, totaling 250 grams of alkali activator. The ratio of sodium silicate to sodium hydroxide was consistently maintained at 2.5 across all mixtures. Through preliminary tests, it was determined that a constant water ratio of 50 grams would be used in the experiments. Additionally, river sand sourced from Sivas Kızılırmak was kept constant at 1350 grams for all mixtures. The prepared samples were produced using a Hobart mixer in accordance with the TS EN 196-1 standard. They were poured into prismatic molds with dimensions of 40x40x160 mm and heat-cured in an oven for 24 hours. After that, the samples were allowed to cure at ambient temperature and kept in airtight conditions for periods of 7, 28, 56, and 90 days, leading up to the experiments.

Blast furnace slag is a by-product generated during the production of iron in blast furnaces at iron and steel manufacturing plants. The rapid cooling of this slag results in an amorphous structure. Granulated blast furnace slag is subsequently ground into a fine powder and used in

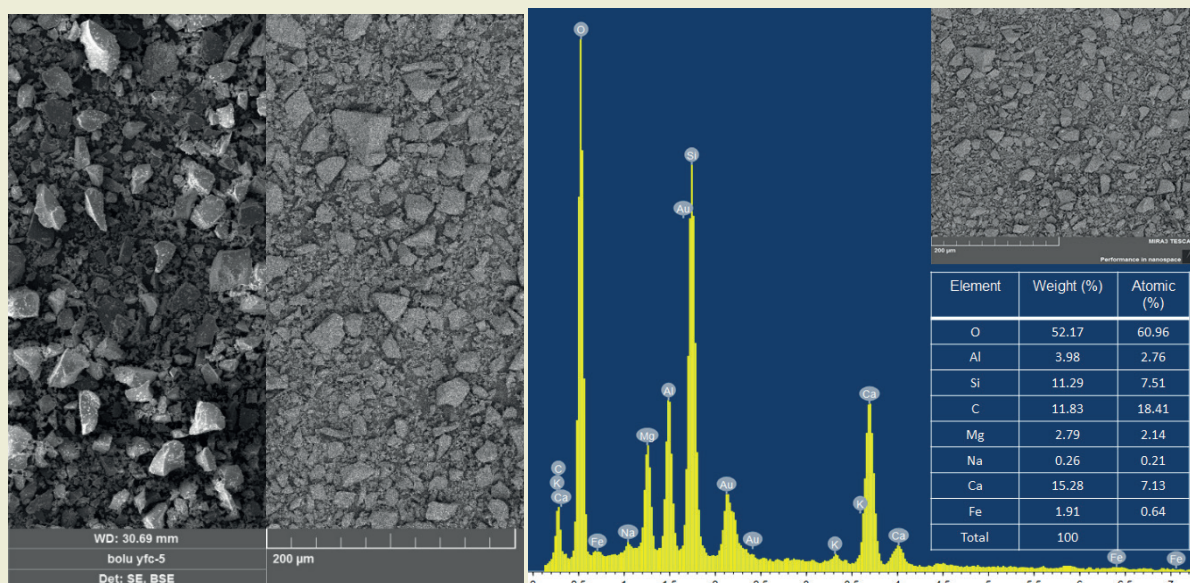


Figure 1. SEM-EDX analysis of Bolu blast furnace slag

the formulation of concrete and mortar. For this study, the slag was procured from Bolu Cement Industry Inc. The silica + alumina + iron oxide ratio is $54.41\% < 70\%$, which is below the minimum percentage requirement for pozzolanic activity [3]. ►Figure 1 presents SEM/EDX analysis of the blast furnace slag, which appears angular, finely structured, and glassy. This fine grain enhances its surface area, offering a potential advantage in terms of reactivity. The physical and chemical properties of the blast furnace slag are detailed in ►Table 1.

Table 1. Physical and chemical properties of Bolu blast furnace slag

Component	(%)
SiO ₂	40.5
Al ₂ O ₃	12.8
Fe ₂ O ₃	1.11
CaO	35.5
MgO	5.81
K ₂ O	0.6
SO ₃	0.22
TiO ₂	0.71
Na ₂ O	0.7
Total	97.95
Specific Surface (cm ² /g)	5384
Moisture (%)	0.1

The Class C fly ash utilized in this experiment was obtained from the Sivas Kangal Thermal Power Plant. This material is classified as Class C (high calcareous) due to its composition, which contains over 50% SiO₂, Al₂O₃, and Fe₂O₃ [14-16]. Additionally, because the CaO content exceeds 10%, these ashes are also referred to as high calcite fly ashes. Typical crystal phases present in Class

C fly ashes include anhydrite (CaSO₄), tricalcium aluminate (Ca₃Al₂O₆), lime (CaO), quartz (SiO₂), periclase (MgO), mullite (Al₆Si₂O₁₃), merwinite (Ca₃Mg(SiO₄)₂), and ferrite ((Mg, Fe)(Fe₃Al)₂O₄). Scanning Electron Microscope (SEM) images of the fly ash reveal a structure that is spherical, glassy, and irregular. ►Table 2 summarizes the physical and chemical properties of the fly ash sourced from the Kangal Thermal Power Plant. ►Figure 2 presents SEM/EDX analysis of the fly ash.

Table 2. Physical and chemical properties of fly ash

Component	(%)
MgO	3.12
Al ₂ SO ₃	14.2
SiO ₂	35.01
SO ₃	7.56
Na ₂ O	1.21
K ₂ O	1.06
CaO	25.75
Fe ₂ SO ₃	5.42
Insoluble residue	24.21
Glow loss	6.02
Total	99.35
Physical composition	
Specific gravity (ton/m ³)	2.72
45 micron sieved balance (%)	44.3
Blain specific surface (cm ² /gr)	3320

Strontium used in the study contains 37.76% SrO as strontium ore in its structure, as waste material obtained from mining sites in the Sivas region. It contains silexite, calcite, mica, and other minerals in its mineralogical composition. The density of the stron-

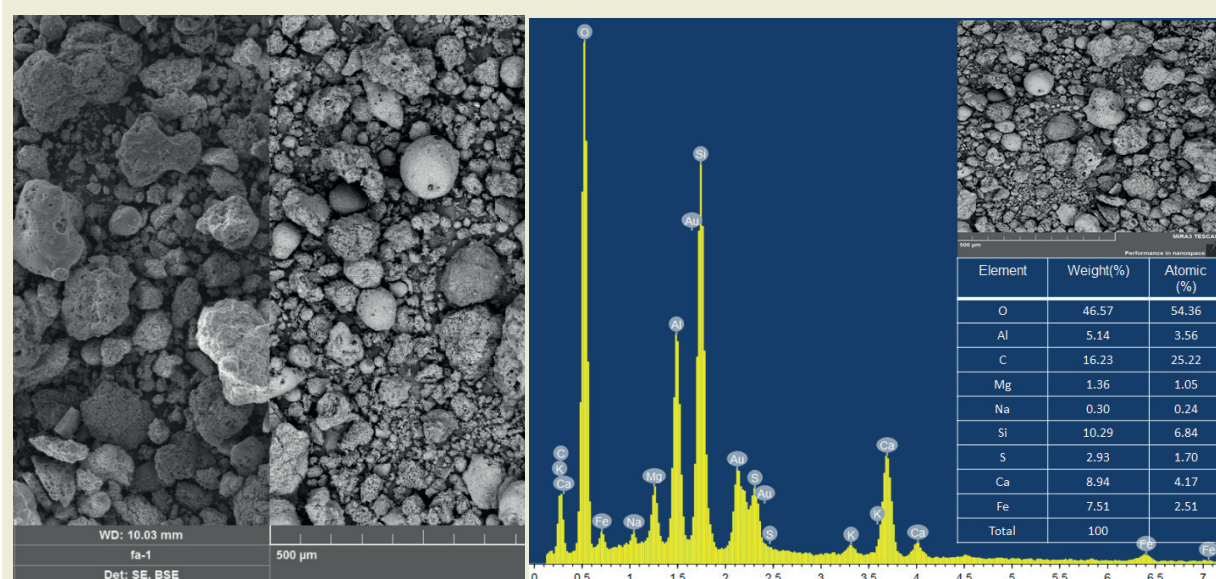


Figure 2. SEM-EDX analysis of fly ash

tium-containing mineral is 3.54 g/cm^3 . ►Figure 3 shows the photographs of the mineral taken before and after grinding. After drying and grinding, it was sieved with the help of sieve number 200, and made ready for use.

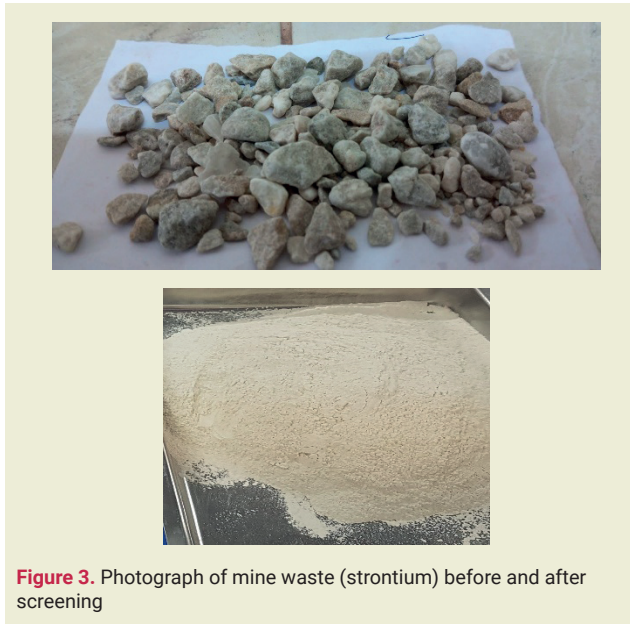


Figure 3. Photograph of mine waste (strontium) before and after screening

The physical and chemical properties of strontium are presented in ►Table 3. ►Figure 4 shows a scanning electron microscope (SEM) image of a strontium mineral at 10.00X magnification. The analysis revealed a grain structure consisting of various sizes and different geometric shapes. Additionally, a semi-quantitative mineral analysis of this mineral was conducted using X-ray fluorescence spectrometry (XRF) at the Erciyes University Technological Research and Application Centre (TAUM), as shown in ►Table 3.

The marble powder employed in the geopolymer composite for this study was sourced as industrial waste

from the Özmersan marble plant in the Sivas region. After drying and grinding, the powder (►Figure 5) was prepared for use by being sifted through a sieve with a mesh size of 200. The marble powder's SEM and EDX analyses, which were prepared for experiments after drying, are presented in ►Figure 6. These analyses were conducted at the Advanced Technology Research and Application Centre (CÜTAM) of Sivas Cumhuriyet University. The results show that the marble powder has a semi-spherical shape and consists of particles of various sizes. Notably, the high CaO ratio in the marble powder's composition stands out during the physical and chemical property assessment. A summary of the physical and chemical properties of the marble powder is provided in ►Table 4.

Table 3. XRF analysis of strontium

Components	%
SrO	37.76
SO ₃	38.63
CaO	14.42
BaO	0.43
SiO ₂	0.18
Al ₂ O ₃	0.084
TiO ₂	0.103
Fe ₂ O ₃	0.091

Titanium dioxide nanoparticles possess specific surface areas ranging from 200 to 220 m^2/g , with particle sizes below 25 nm and melting temperatures of approximately 1825 °C [17]. TiO₂ exhibits three distinct crystal structures: rutile, anatase, and brookite [18]. The titanium dioxide used in these experiments was sourced from the Mega Analitik firm. Scanning Electron Microscopy (SEM) images illustrating the properties of TiO₂ are provided in ►Figure 7. These images reveal

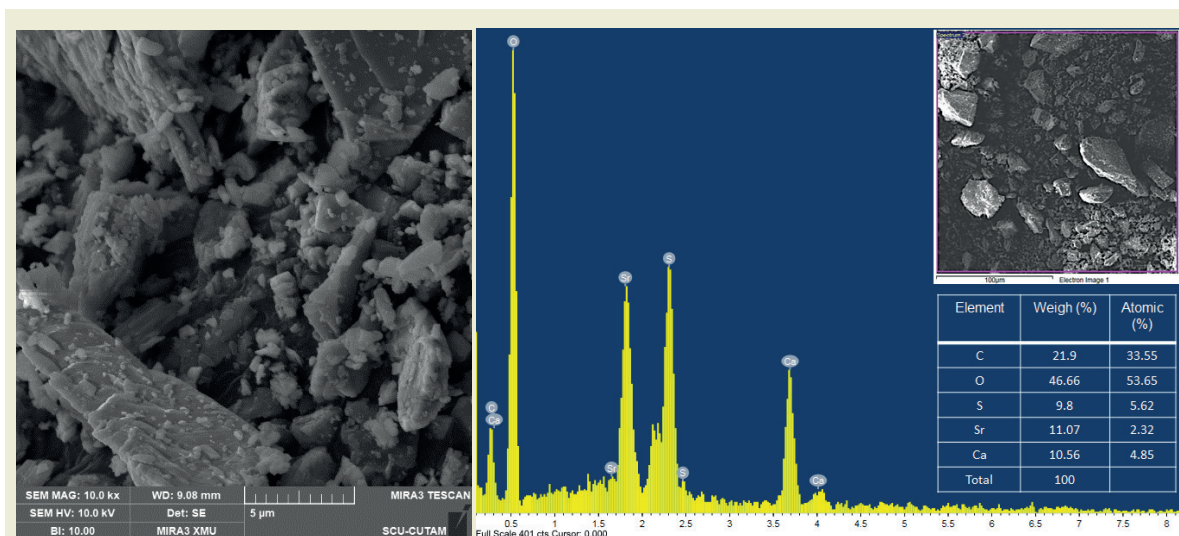


Figure 4. SEM-EDX analysis of mine waste /strontium



Figure 5. Photograph of sifted marble powder

that TiO_2 has a crystalline structure and predominantly spherical shapes.

Table 4. Physical and chemical properties of marble powder

NOCHE - Travertine Components	%
Al_2O_3	0.22
CaO	54.51
FeO_3	0.11
MgO	0.41
SiO_2	0.87
Density (g/cm^3)	2.69

3. Results and Discussions

The flexural and compressive strengths of the specimens at the end of the curing period are presented in ►Figure 8 and ►Figure 9, respectively. This figure il-

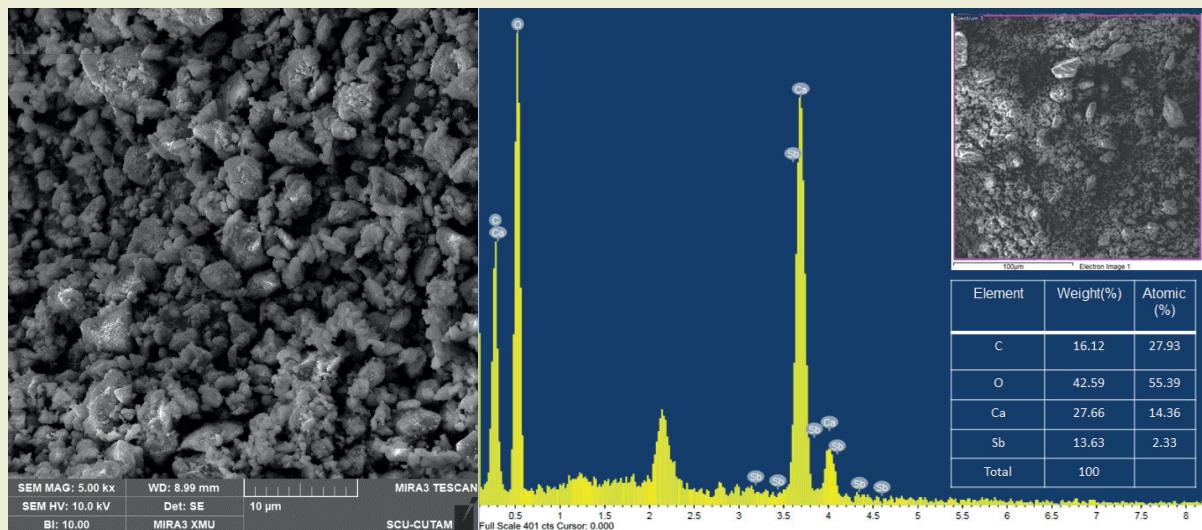


Figure 6. SEM-EDX analysis of marble powder

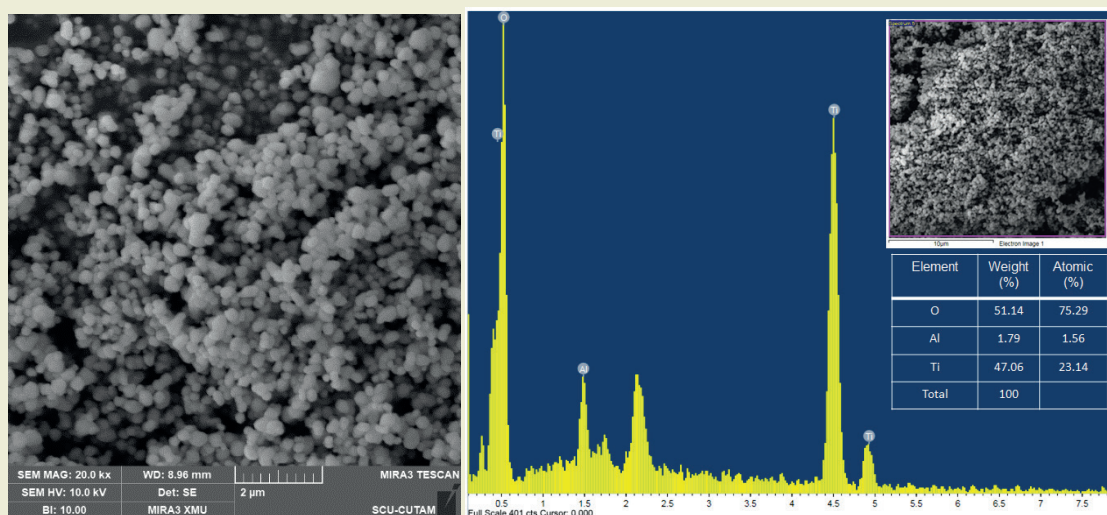


Figure 7. SEM-EDX analysis of titanium dioxide

illustrates the flexural strength results for samples cured for 7, 28, 56, and 90 days. It shows a time-dependent decrease in flexural strength. The highest flexural strength value, 10.81 MPa, was observed in the strontium-doped sample at the 7-day mark, while the lowest flexural strength, 7.66 MPa, was found in the titanium dioxide-doped sample at 90 days. Upon examining the compressive strengths, the highest value recorded was 65.7 MPa for titanium dioxide in the 7-day samples. Conversely, the lowest compressive strength, measured at 51.5 MPa, was observed for strontium in the 90-day samples. Overall, it is evident that titanium dioxide exhibits the highest compressive strength among the various additives tested. This indicates that the use of

a low concentration of 1% titanium dioxide positively influences compressive strength [19].

The SEM/EDX images of the mortars, produced by substituting 5% marble dust for blast furnace slag and fly ash as the primary binder, are presented in ►**Figure 10** for both the 7-day and 90-day samples. Analysis of the SEM/EDX images for the 7-day samples indicated a Si/Al ratio of 4.02. In comparison, the 90-day samples revealed a Si/Al ratio of 3.92. This reduction in the Si/Al was consistent with a decline in compressive strength. When the compressive strengths of 7 and 90-day samples are examined, it is thought that the decrease in compressive strength seen in 90-day samples

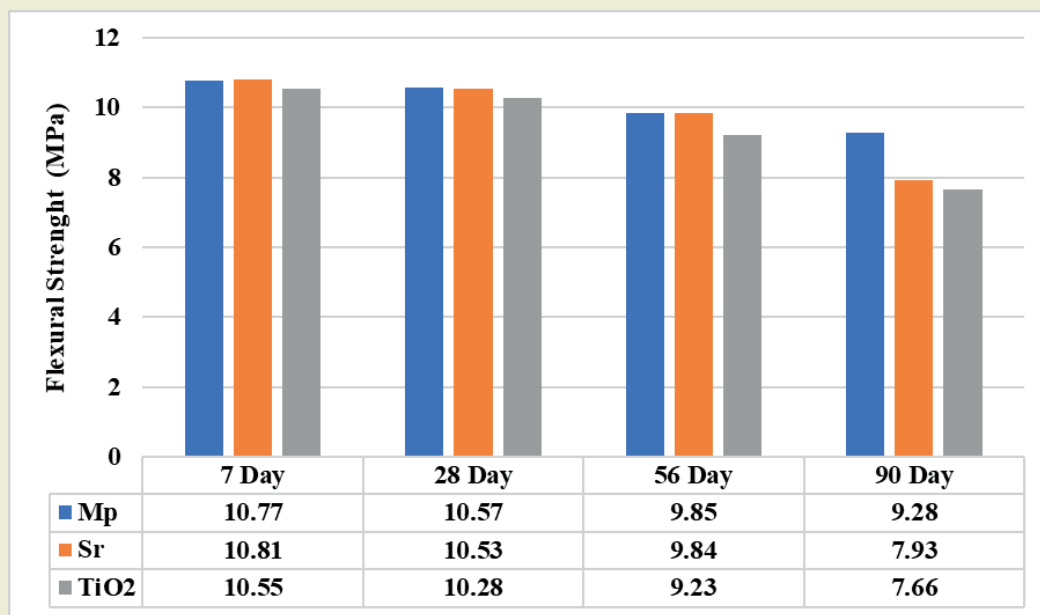


Figure 8. Flexural strengths of marble powder, strontium, and titanium dioxide

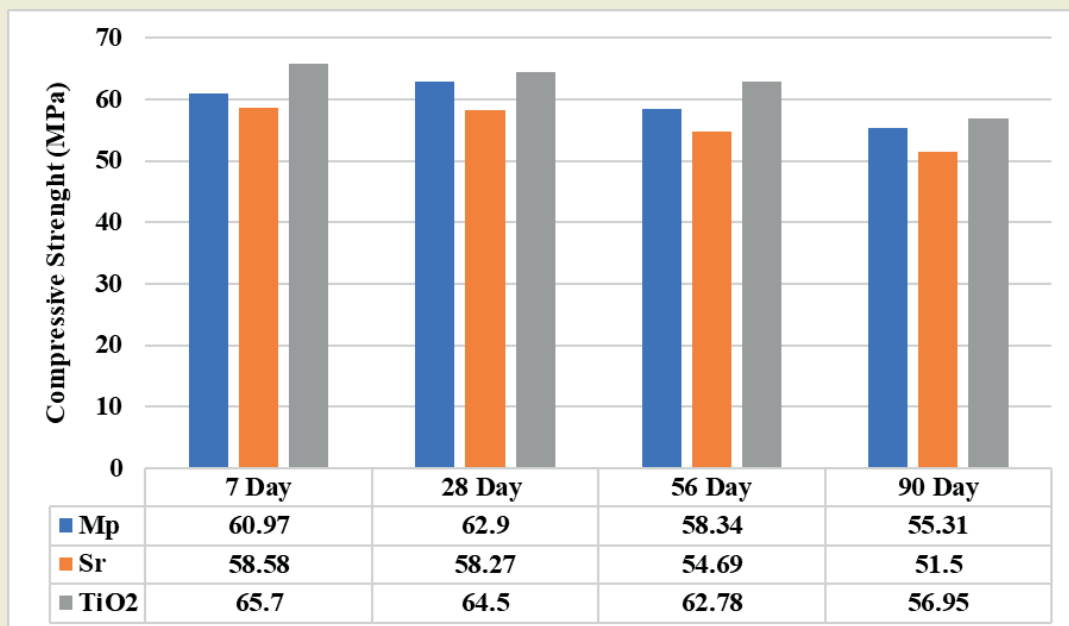


Figure 9. Compressive strengths of marble powder, strontium, and titanium dioxide

may be due to the decrease in Si/Al ratio. In addition, when SEM images were examined, it was determined that a more homogeneous structure was formed in the 7-day sample images, but unreacted particles were formed in the 90-day sample images. When 7 and 90-day compressive strengths are compared, it is thought that the decrease in compressive strength in 90-day samples may be due to the decrease in Si/Al ratio. In addition, when SEM images are examined, it is predicted that a homogeneous structure is formed in 7-day images and unreacted particles are formed in 90-day images, which may be effective on strength. It has been stated that the relatively high compressive strength of binders with high slag content is due to the density of C-A-S-H gel phases and microstructure [20-21]. On 90-day specimens, waste materials enhance microstructure densification and contribute to early strength, but may reduce strength later. [22]. It could be said that the marble powder additive is effective on the strength with its void reducing effect and Ca content. Thanks to the

CaO content of marble powder, the effect of gelation on microstructural analyses is observed [23].

► **Figure 11** shows SEM and EDX images of geopolymer mortars that include a 5% substitution of strontium in a mixture of blast furnace slag and fly ash. In the Energy Dispersive X-ray (EDX) analysis of 7-day geopolymer mortars, the silicon to aluminum (Si/Al) ratio was found to be 3.16. In contrast, the EDX analysis of 90-day geopolymer mortar samples revealed a Si/Al ratio of 5.75 on the surface. Upon examining the Scanning Electron Microscopy (SEM) images, a denser Calcium-Silicate-Aluminate-Hydrate (C-S-A-H) gel formation was observed in the 7-day geopolymer mortars with 5% strontium doping, despite the presence of micro-cracks. Similar gels were noted in the 90-day geopolymer mortars with 5% strontium doping, but it was also observed that micro-cracks appeared, particularly between the layers, along with the presence of unreacted particles.

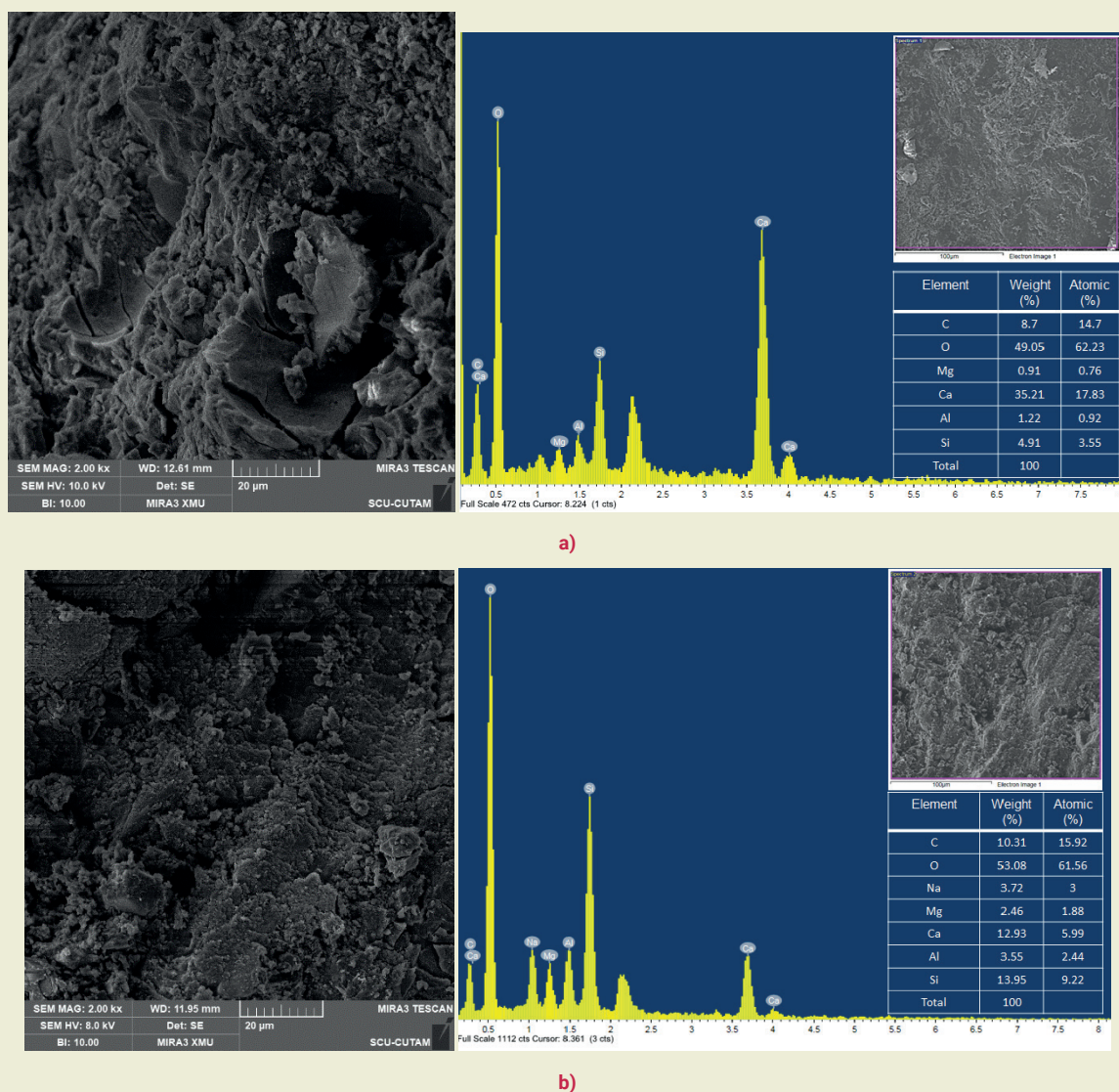


Figure 10. SEM (left) /EDX (right) images of 7 days (a) and 90 days (b) old marble powder-doped geopolymer mortar

The SEM/EDX images of geopolymer mortars with 1% titanium dioxide substitution are presented in ►Figure 12. The Si/Al ratio was found to be 4.76 in the samples taken after 7 days, and 15.92 in those taken after 90 days. Examination of the SEM images revealed that the 7-day geopolymer mortar samples exhibited a more homogeneous internal structure compared to the 90-day samples, leading to a better bond among the components of the matrix.

4. Conclusions

In the geopolymer mortars designated as 85BFS15FA, the composition included 5% marble powder, 5% strontium, and 1% titanium dioxide. The samples were cured in an oven for 24 hours with a 10M NaOH solution at a temperature of 75°C. The findings revealed a reduction in both flexural and compressive strengths as curing time increased. Remarkably, titanium dioxide demonstrated the highest compressive strength at early ages

(specifically at 7 days). Scanning Electron Microscopy (SEM) analyses indicated that the geopolymer mortars with titanium dioxide developed gel structures, which are thought to enhance compressive strength. This effect is likely attributed to the high proportion (85%) of blast furnace slag combined with the low proportion (1%) of titanium dioxide. It was observed that TiO_2 additive, which was effective on compressive strengths, decreased flexural strengths. Especially for all additives, while the flexural strengths of the 7 and 28 day cured specimens were similar, time-dependent decreases in these values were observed.

The results of this study show that it can contribute to the recycling of local and regional wastes and sustainability issues. In addition, by presenting 3 different additives comparatively, the mechanical and microstructural differences were compared. The fact that a high strength result was obtained at early age with TiO_2 admixture at a low rate of 1% in the study showed that it can be used for low performance admixed geopolymer

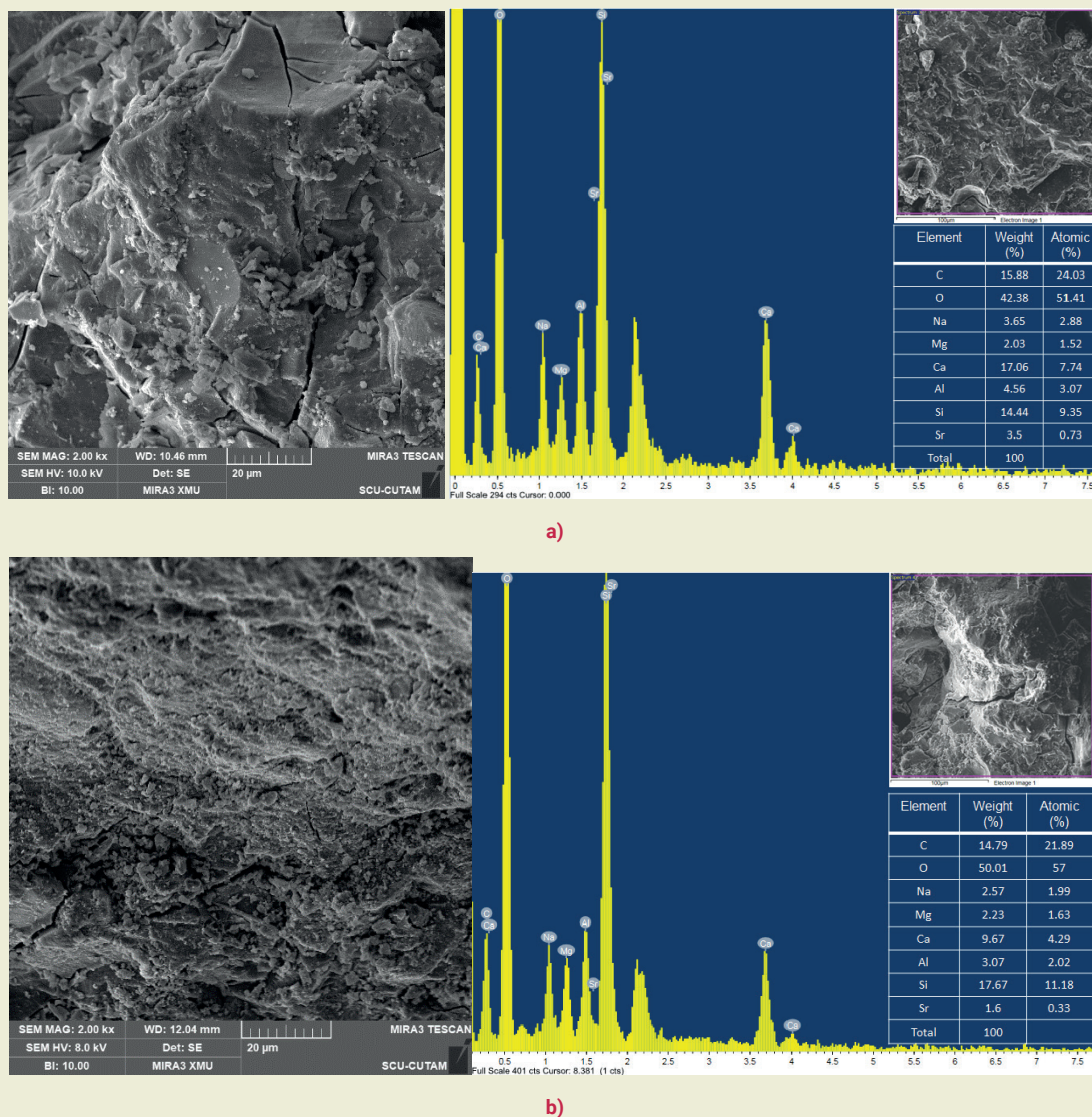


Figure 11. SEM (left)/EDX (right) images of 7 days (a) and 90 days (b) strontium-doped geopolymer mortar

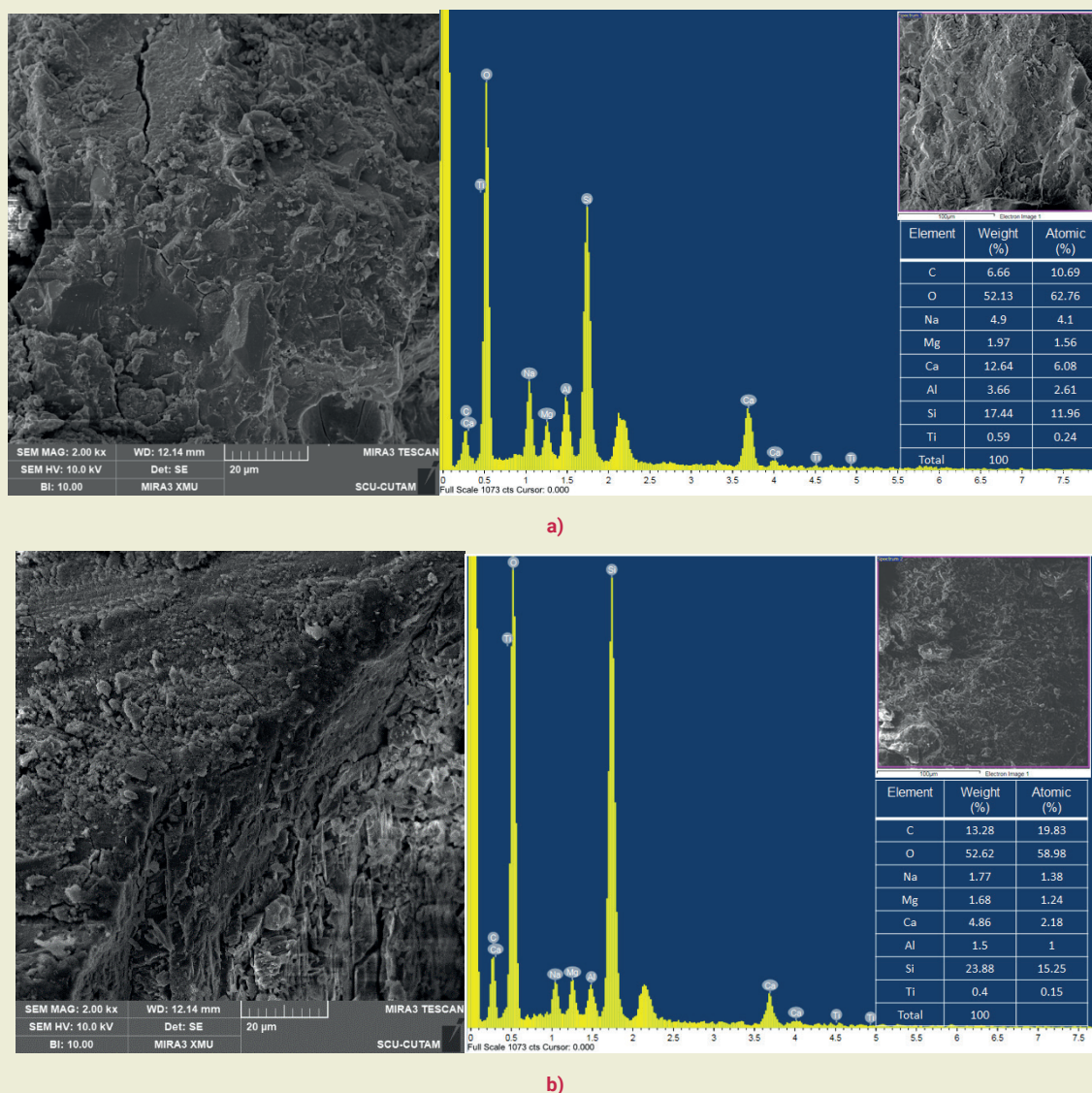


Figure 12. SEM (left)/EDX (right) images of 7 days (a) and 90 days (b) TiO₂ doped geopolymer mortar

concrete and mortars.

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Research ethics

Not applicable.

Author contributions

Conceptualization: [Hülya Temizer, Özlem Çavdar], Methodology: [Hülya Temizer, Özlem Çavdar], Formal Analysis: [Author names], Investigation: [Hülya Temizer, Özlem Çavdar], Resources: [Hülya Temizer], Data Curation: [Hülya Temizer], Writing - Original Draft Preparation: [Hülya Temizer, Özlem Çavdar], Writing - Review & Editing:

[Özlem Çavdar], Visualization: [Hülya Temizer, Özlem Çavdar], Supervision: [Özlem Çavdar], Project Administration: [Özlem Çavdar], Funding Acquisition: [Hülya Temizer, Özlem Çavdar]

Competing interests

The authors state no conflict of interest.

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Data availability

The raw data can be obtained on request from the authors.

Peer-review

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