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Research Article

An Investigation of the Effect of Waste Glass Additive on Strength and Microstructure Properties of Mortars

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ABSTRACT

The importance of recycling waste in terms of ecological efficiency becomes more evident every day. Cement based mortars are one of the building materials that are widely used in building production. Especially the energy and raw materials consumed in cement production are important in terms of ecological efficiency. Savings in the amount of cement in cement-based mortars will be beneficial in this sense. With this perspective, in this study, the usability of waste glass in mortar production was investigated experimentally. Following the production stage of the test specimens, workability measurements were made first. Compressive and flexural strength tests were performed on the produced mortar samples to determine their strength properties. Microstructural examinations were carried out using the SEM technique. Unlike other studies, morphological findings were detailed with the help of EDX analyses. As a result, with the addition of waste glass, a decrease in the strength values of the test specimens was determined. It was also found that glass wastes with 10% substitution rate improved the workability.

Keywords: *Waste glass, Microstructure, Strength*

Cam Atıkların Harçların Dayanım ve Mikroyapı Özelikleri Üzerine Etkisinin Araştırılması

ÖZ

Ekolojik verimlilik açısından atıkların geri dönüştürülmesinin önemi her geçen gün daha da belirginleşmektedir. Çimento esaslı harçlar, yapı üretiminde yaygın olarak kullanılan yapı malzemelerinden biridir. Özellikle çimento üretiminde tüketilen enerji ve hammadde ekolojik verimlilik açısından önemlidir. Çimento esaslı harçlarda çimento miktarında yapılacak tasarruflar bu anlamda faydalı olacaktır. Bu bakış açısıyla, bu çalışmada atık camın harç üretiminde kullanılabilirliği deneysel olarak araştırılmıştır. Test numunelerinin üretim aşamasını takiben öncelikle işlenebilirlik ölçümleri yapılmıştır. Üretilen harç numuneleri üzerinde basınç ve eğilme dayanımı testleri gerçekleştirilerek dayanım özellikleri belirlenmiştir. Mikroyapı incelemeleri SEM tekniği kullanılarak gerçekleştirilmiştir. Diğer çalışmalardan farklı olarak EDX analizleri yardımıyla morfolojik bulgular detaylandırılmıştır. Sonuç olarak, atık cam ilavesi ile test numunelerinin dayanım değerlerinde düşüş tespit edilmiştir. Ayrıca %10 ikame oranına sahip cam atıklarının işlenebilirliği artırdığı tespit edilmiştir.

Anahtar Kelimeler: *Atık cam, Mikroyapı, Dayanım*

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I. INTRODUCTION

Construction wastes that arise due to the increase in the world population, industrial progress and the changes in the construction sector create an ecological danger in general, but also an urgent need for recycling [1]. In this sense, the construction industry, together with its many production branches, can offer great advantages in terms of the use and production of recycled materials [2].

Sodium-calcium-based glass wastes from crushed glass waste or building debris can be recycled again in the construction industry. The point to be considered here is to pay attention to the situation that the glass can be recycled again in demolition techniques. Particular attention should be paid to this point in glass waste that can be recycled in the form of aggregates and additives [3-7].

Although the glass does not experience any quality loss after the recycling process, a large amount of glass waste remains unrecyclable [8]. In Turkey, 1,608,669 tons of glass were produced in 2019 and 871,426 tons were put on the market. The recycled amount is 276,037 tons and the recycling rate is approximately 32% [9].

Different materials are used as cement substitute materials in construction materials such as cement-binding based mortar and concrete. Glass waste can also be evaluated within the scope of these substitute materials. However, the most important point to be considered here is the expectation of alkali-silica reaction originating from waste glass and aggregate [10]. However, when the literature data is examined, it is stated that this issue does not occur if the waste glass is finely ground under 75 microns [11]. In addition, no adverse effects were observed in the strength and durability of the mortar samples with waste glass additive up to 20% compared to the samples without additives [12].

Experimental and literature reviews on the use of waste glass as a cement replacement material have been investigated by various studies [13-21]. Salim and Morpanah [22] focused on the curing regime at different temperature levels in their studies, in which they replaced waste glass with a maximum of 20% cement. They state that the 10% substitution rate is suitable for use. Pachideh et al. [23], on the other hand, determined that there was a positive increase in mechanical properties as a result of their experimental study with waste glass, which they replaced with a maximum of 28% cement. Younes et al. [24], on the other hand, stated that the mechanical properties of the waste glass additive mortars at 20% cement substitution rate improved positively.

In line with this information, the aim of this study is to focus on the morphology and strength analysis of mortars replaced with 10%, 20% and 30% waste glass. The microstructures of the samples were analyzed using SEM and EDX techniques. In addition, physical and mechanical property findings were tried to be explained in this context.

II. MATERIALS and METHODS

A. MATERIALS

In this study, CEM II/A-M (P-LL) 42.5 R type Portland cement conforming to EN 197-1 [25] was used. Chemical components of cement and waste glass are given in Table 1. For the preparation of mortar mixes, CEN standard sand in accordance with EN 196-1 [26] was used.

The waste glass used in this study was soda-lime based glass obtained from transparent glass bottles. The collected waste glass was first crushed coarsely, then the grinder was ground into powder (Figure 1).

Figure 1. *Glass waste powder*

The graph of laser particle size analysis of the obtained waste glass is given in Figure 2. The laser grain size analysis values for the glass powder (GP) were determined as d_{10} = 2.504 µm, d_{50} = 26.62 µm, and d_{90} = 98.54 µm with an average particle size of 68 µm.

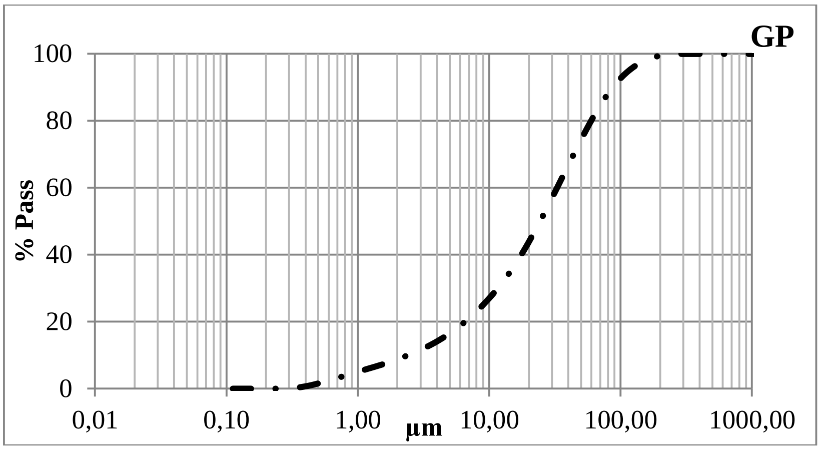


Figure 2. *The particle size analysis of glass waste powder*

The pozzolanic reactivity behavior of GP, which improves the mechanical and durability properties of concrete, is significantly influenced by the GP fineness [27]. XRD graph of the waste glass was examined, it was determined that the peak region between 20-30 Theta indicates that the waste glass powder has an amorphous structure. (Figure 3).

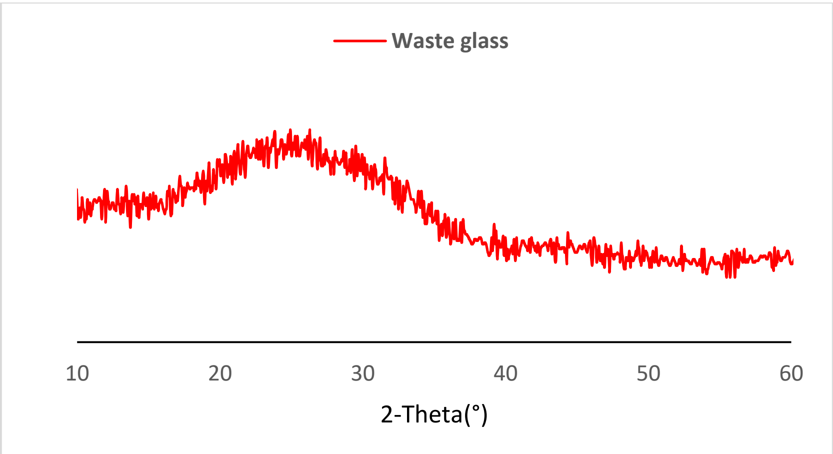


Figure 3. *XRD graph of glass waste powder*

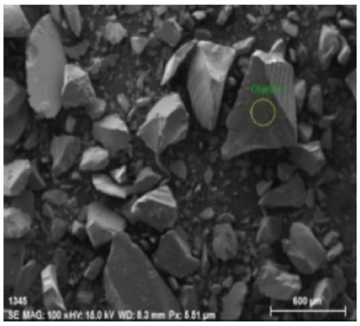
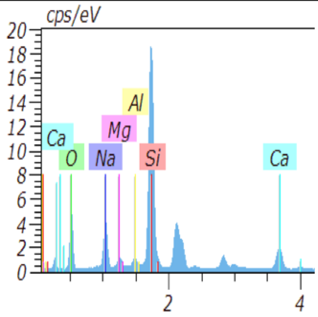
<table>
<tr><th colspan="2">Waste glass</th><th>Elements</th><th>Wt%</th></tr>
<tr><td rowspan="6"></td><td rowspan="6">
</td><td>Si</td><td>44.74</td></tr>
<tr><td>O</td><td>31.23</td></tr>
<tr><td>Ca</td><td>11.27</td></tr>
<tr><td>Na</td><td>9.44</td></tr>
<tr><td>Mg</td><td>1.87</td></tr>
<tr><td>Al</td><td>1.45</td></tr>
</table>

Figure 4. *SEM-EDX analysis of GP*

In previous studies [28, 29], waste glass powder had a silica content of 59–75%, and it was similar in this study (Figure 4).

B. METHODS

B. 1. Sample Preparation

The EN 196-1 [26] standard was used in the preparation of the mortar. Mortars were then prepared in accordance with this standard using the following mixing procedure: firstly, for mortar production, water is first put into the mixer, which is then operated at low speed for 30 seconds while adding cement, then, CEN standard sand addition in the next 30 seconds is done, the mixer is run at high speed for 30 seconds, after 90 seconds, the device is stopped, the mortar adhering to the rim of the mixing bowl is mixed, and production is continued at high speed for another 60 seconds, and finally the production process is completed. The mix proportions of the studied mortars are summarized in Table 1.

Table 1. *Mixture proportions*

Mix	**Binders**		**Binder/Aggregate**	**W/B**	**Water**	**Paste volume (%)**
	Cement	GP				
Control	1		1/2.7	0.5	0.45	36.79
GP5	0.95	0.05	1/2.7	0.5	0.45	36.97
GP10	0.90	0.1	1/2.7	0.5	0.45	37.16
GP15	0.85	0.15	1/2.7	0.5	0.45	37.34

B. 2. Flow Table Test

The flow indicates the workability of the sample. For this purpose, a flow test according to ASTM C1437 [30] was performed to evaluate how the substitution of waste glass powder in the mortars affected the flow. For this test, mortar samples were placed inside a truncated cone that was positioned in the middle of the flow table. The flow table was dropped continuously for 25 times in 15 seconds after the mold had been slowly removed. The flowability index (Γ) values, as described by Kim et al. [31], were calculated for each mortar mixture using the spread values measured after the test, using the following formula (1):

$$\Gamma = \frac{\mathrm{d}_1 . \mathrm{d}_2 - d_0^2}{d_0^2} \tag{1}$$

where, d_0 is the base internal diameter of the mold (100 mm), d_1 is the largest spread diameter, and d_2 is the diameter perpendicular to d_1(Figure 5).

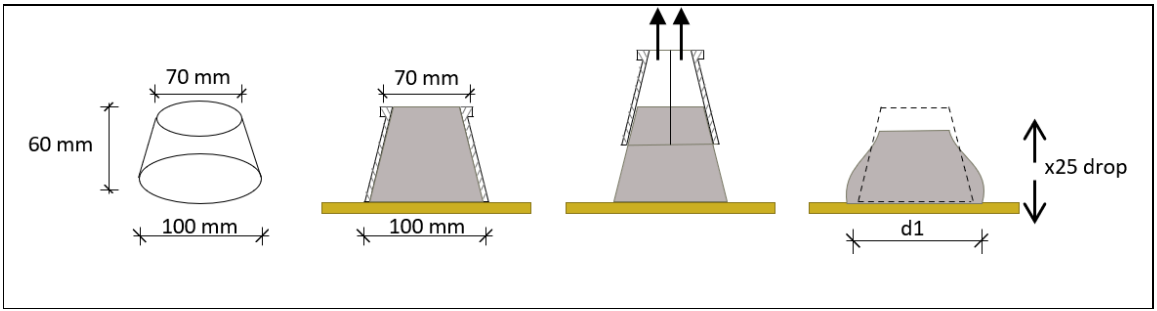


Figure 5. *Flow table test*

B. 3. Compressive and Flexural Strength Tests

Using 7, 28, and 56 day samples of each mortar in accordance with EN 1015-11 [32], the compressive strength and flexural tensile tests were assessed in order to acquire and compare the mechanical properties. Prismatic samples (40 × 40 × 160 mm) for each mortar were poured homogeneously and placed with a shaking table. The specimens were demolded approximately 24 hours after casting and then cured in the curing tank at 20 ±2 °C before being tested in lime-saturated water.

B. 4. Microstructure Investigations

To evaluate the microstructure and surface morphology of hardened mortar samples, SEM and EDX analyses were performed using a scanning electron microscope (SEM). The samples were oven dried and coated with Au-Pd before imaging by the instrument supervisor. The samples were oven dried and coated with Au-Pd by the instrument supervisor, and images were taken using a Zeiss Evo10 device images were taken using a Zeiss Evo10 device at an EHT voltage of 15 kV with magnifications ranging from 7X to 100,000X

III. RESULTS and DISCUSSION

A. FLOW TABLE TEST RESULTS

Figure 6 presents the influence of waste glass powder on the workability of the mortar mixtures. It is noticed that the fresh Control mortar mixture presents a slump flow of 17.33 cm; when 5%, 10%, or 15% of the cement was replaced by waste glass powder, the slump flow was 17.50 cm, 18.33 cm, and 16 cm, respectively. As seen in Figure 6 an increase in spreading values was observed with a 5% and 10% substitution of waste glass powder in cement [33-35]. However, there was a decrease in workability with the 15% glass powder substitution to cement. Similar results were reported by Salim et al. [32]. The substitution of glass powder to cement above a certain ratio (15% for this study) reduced the rolling due to the angular shape of the glass powder particles compared to cement, and the cohesiveness of the mixture decreased due to the decrease in the amount of cement. Accordingly, as a result of the decrease in the lubricating effect, the spread of the mixture remained low, while the material accumulation was higher.

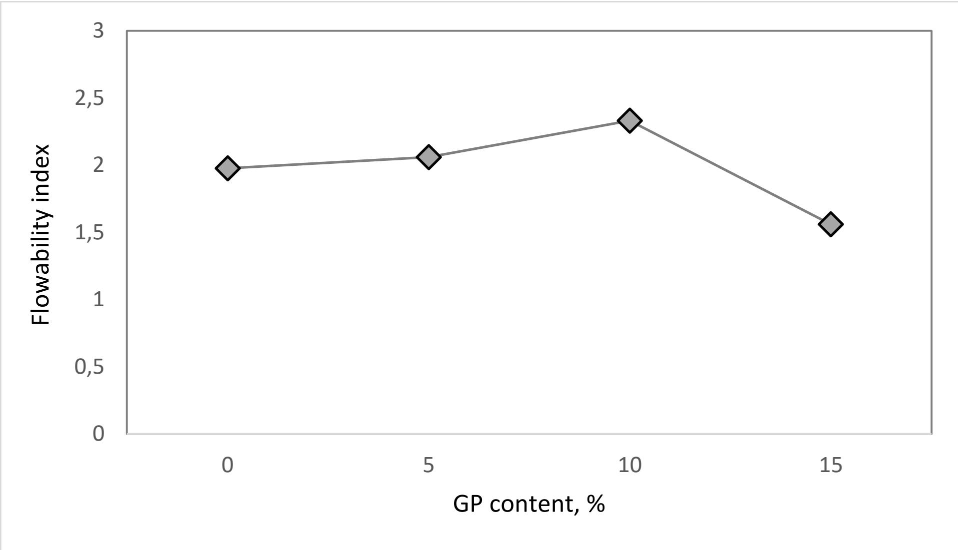


Figure 6. *Flowability index results*

B. COMPRESSIVE and FLEXURAL STRENGTH RESULTS

Figure 7 shows the 7, 28 and 56-days compressive strengths of mortar samples. The use of waste glass powder replaced by cement led to a decrease in compressive strength at all curing ages and replacement ratios. This reduction in strength compared to the control mixture is more prominent at the 7-day samples than at the later ages (28- and 56-day). As seen in Figure 7, at 7 days, with the increase in GP content (5%–15%), the strength activity decreased (19.8%–39.8%). For 28 days, the reductions in strength activity index were 15%, 7.4%, and 21.2%, while for 56 days they were 9.7%, 12%, and 19.2% for using GP 5%, 10%, and 15%, respectively. This is due to increased C-S-H gels and enhanced Interfacial Transition Zone (ITZ) bond strength as a result of the pozzolanic reaction occurring at a later stage [36-40]. Table 8 shows that the porosity decreases with later ages (28 and 56 days). Among the mixtures containing waste glass powder, the GP5 mixtures had the highest compressive strength at the curing ages of 7 and 56 days, while it was the GP10 mixtures at 28 days. Figure 7 shows the flexural tensile strengths of mortars at 7, 28 and 56-days. The flexural tensile strengths of the samples varied from 6.37 to 11.2 MPa for all curing days. In general, with the increase in the waste glass powder substitution ratio, a decreasing trend was observed in the flexural tensile strengths. With %5, 10% and 15% glass powder substitution, there was a 9.9%, 19.4%, and 26.8% reduction in flexural tensile strengths compared to the control mixture for 7 days, respectively. For 28 days, there was a 10.4%, 9.3% and 20.3% reduction in flexural strengths. For 56 days, there was a 11.6%, 9.6% and 15.9% reduction in flexural strengths.

It can be concluded from these results that as the pozzolanic reactions developed, the flexural tensile strength reduction rates decreased compared to the control mixture. On the other hand, in the 7-day samples in which the pozzolanic reactions did not occur completely, the ratio of decrease in strength increased with the increase in the glass powder replacement rate. For the ultimate strength, the optimum waste glass powder substitution ratio in flexural tensile strength was determined to be 10%.

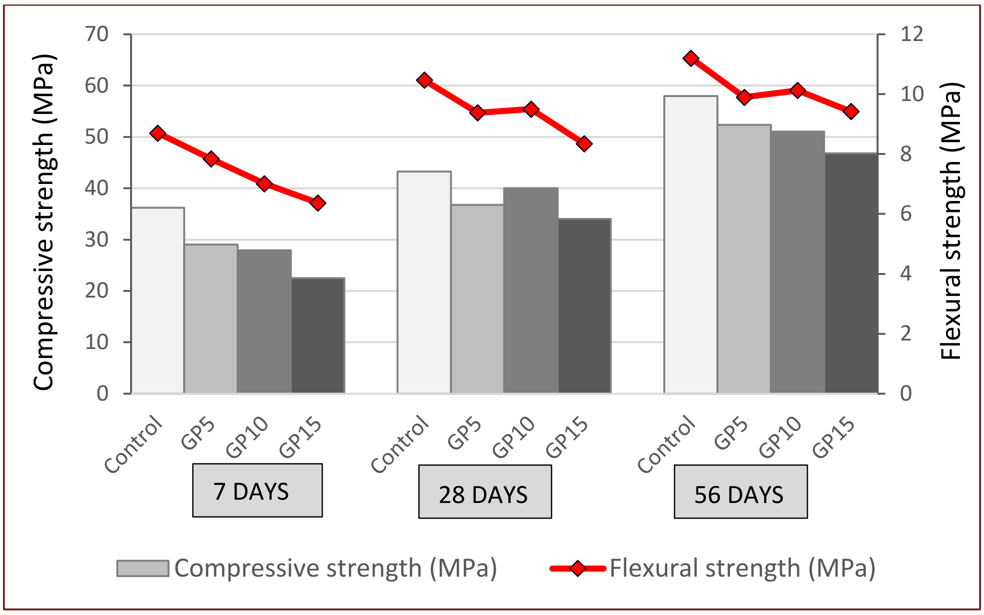


Figure 7. *Compressive and flexural test results*

B. MICROSTRUCTURE ANALYSIS RESULTS

The scanning electron microscope (SEM) was performed to investigate the impact of glass powder substitution at various ratios on the microstructure of mortar samples. The SEM and EDX results are displayed in Figure 8 and 9 respectively. It has been reported that darker regions in SEM pictures contain more hydrated components than lighter regions [41]. Figure 8 shows that the 28- and 56-day samples are more densely structured than the 7-day samples.

In the SEM images of the 7-day samples, fibrous products formed during the early hydration stage are seen, while in the 56-day samples, crumpled foils and dense inner products are dominant. Additionally, the samples' fibrillar morphology transformed into a crumpled morphology as the glass powder ratio grew. With the reaction of the pozzolanic material and the micro-filler effect of the glass powder additive, a dense structure is observed in the GP15 sample as shown in Figure 8 (i).

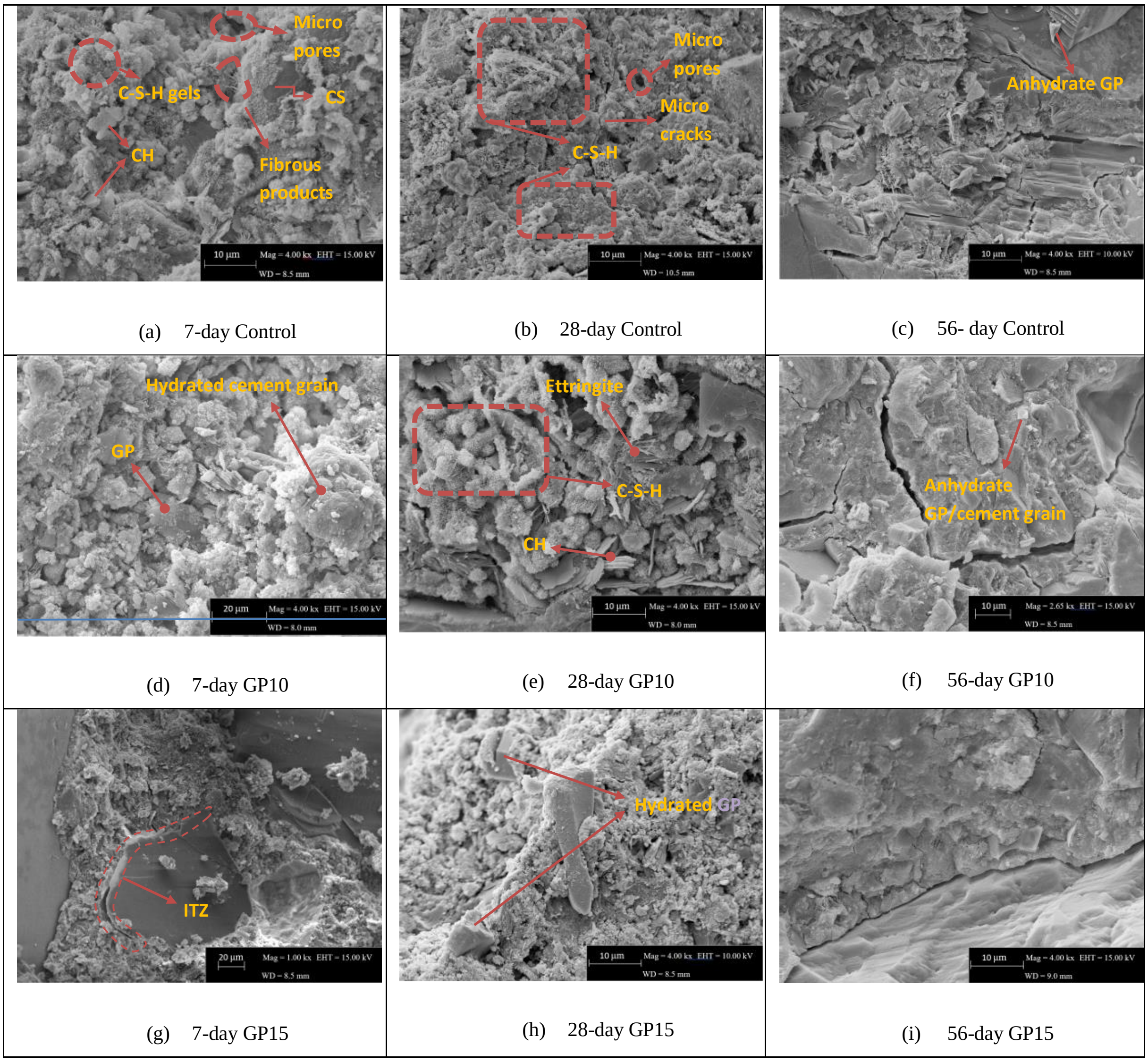


Figure 8. *SEM analysis results*

As seen in Figure 8 (f) and (i) that microcracks became more widespread as the percentage of glass powder in the 56-day samples increased. The negative effect of microcracks on the adherence between the matrix and aggregate phase was also reflected in the decrease of flexural strengths. The microstructures depicted in Figure 8 illustrate the internal composition of the incompletely hydrated paste. As the process of hydration advanced, the interior structure underwent gradual filling with hydration products. Concurrently, the occurrence of pozzolanic reactions led to the consumption of calcium hydroxides and the subsequent formation of a progressively denser texture.

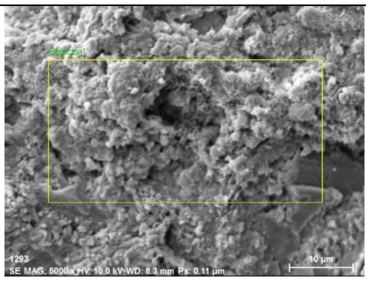
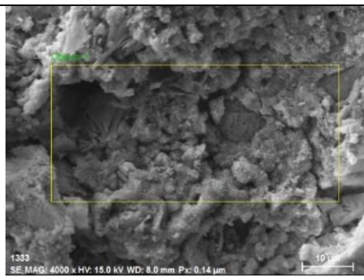
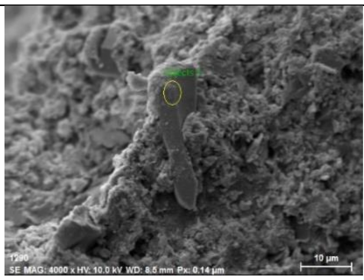
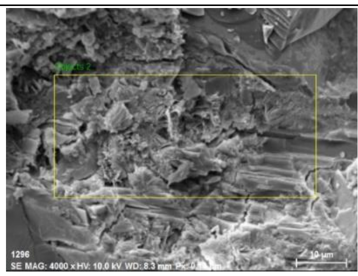
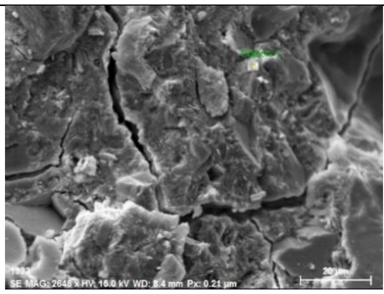
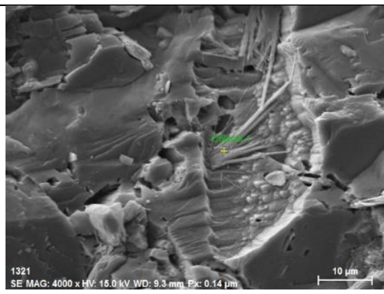
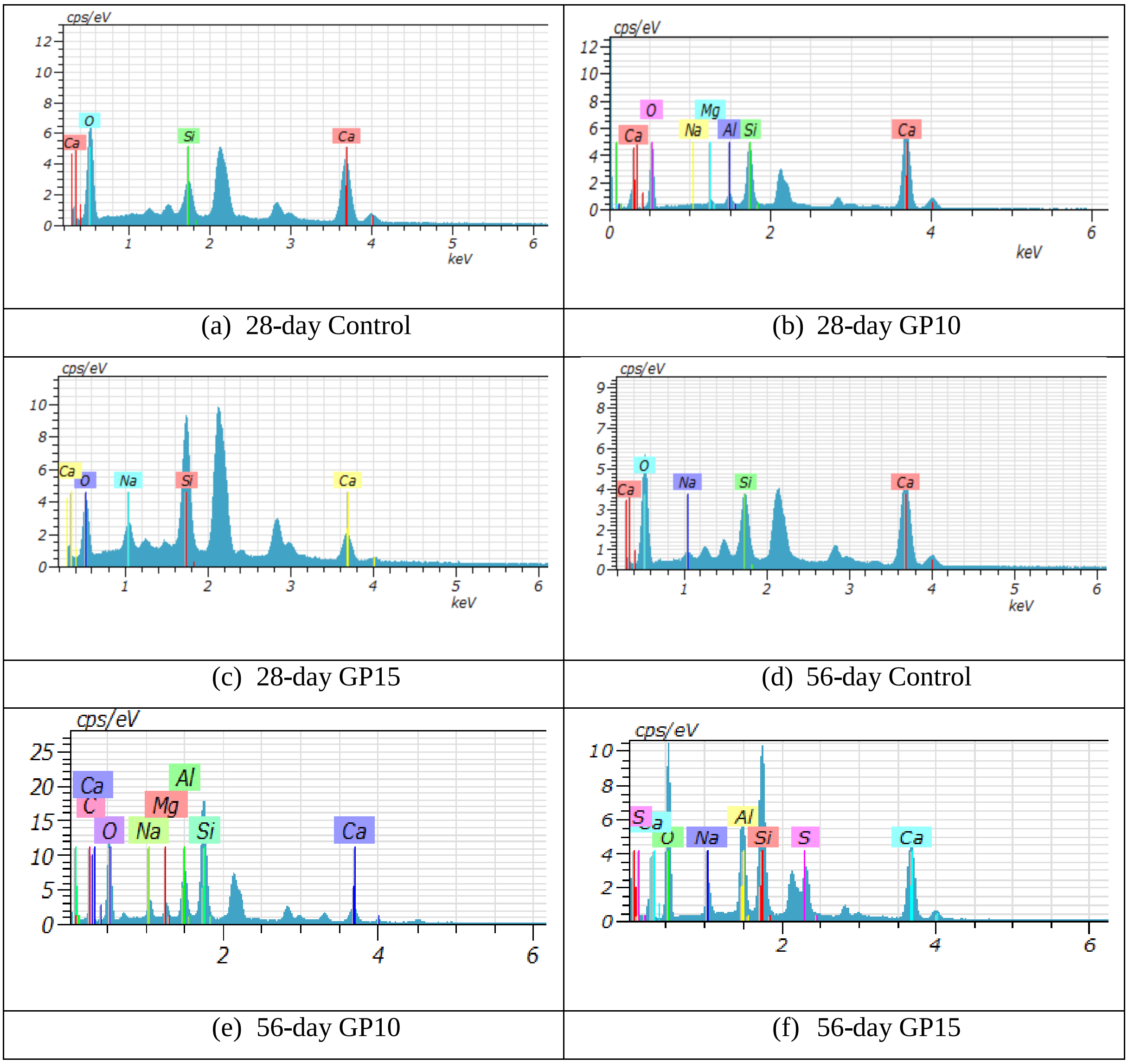
	
(a) 28-day Control	(b) 28-day GP10
	
(c) 28-day GP15	(d) 56-day Control
	
(e) 56-day GP10	(f) 56-day GP15

Figure 9. *SEM-EDX images of test samples*

Energy-dispersive X-ray (EDX) analysis were used to examine the characteristics of the hydrated binder. 28 and 56 days of control, 10% and 15% glass powder containing samples are given Figure 9,10. EDX analyzed regions are marked on the SEM images. As the curing age of the specimens containing glass powder increased, there was a significant decrease in Ca/Si ratios compared to the control specimen. The reasons for this decrease are likely to be the high silica content of glass powder and the consumption of portlandite as a result of pozzolanic reactions. Some studies [42], [43] have reported an increase in C-S-H voids as well as high drying shrinkage with decreasing Ca/Si ratio. A similar finding was also observed in this study. The SEM image of the 56-day GP10 sample with the lowest Ca/Si ratio (0.97) shows an excess of cracks (Figure 10 (e)). Mg peaks in the EDX spectrum may be a marker of developing MgO. The internal stresses caused by the MgO component, which has a high expansion capacity, may have increased the crack formation. Additionally, Mg suggests the development of an alkali-activated gel. The low Ca/Si ratio generally favors the binding of alkalis by CSH and CASH gels [44]. In this study, as seen in the spectrum results of the samples containing glass powder, Na ions are observed which are not observed in the control mixtures. This may support the argument that C-S-H and C-A-S-H gels prevent ASR formation by binding alkali ions.



(a) 28-day Control
(b) 28-day GP10
(c) 28-day GP15
(d) 56-day Control
(e) 56-day GP10
(f) 56-day GP15

Figure 10. *EDX results of test samples*

IV. CONCLUSION

By replacing waste glass with cement in mortar production, the following results were achieved;

- it was found that the compressive and flexural strength values of the mortar samples produced decreased compared to the control samples.
- In the fluidity values, which is an important property on fresh mortars and directly affects the workability, an increase in the fluidity index values was observed with the increase in the substitution rate of waste glass admixture. The highest viscosity values were obtained in the series where waste glass additive was used with 10% replacement rate.
- It has been observed that the development of micro cracks increases due to the increase in the amount of waste glass powder in the mortar, and therefore cement paste aggregate adherence is negatively affected by this situation.
- In the following hydration days, a significant decrease in Ca/Si ratios was detected depending on the silica content of the GP.
- It was observed that due to the increase in the waste glass additive ratio, the cement paste density existing in the microstructure was negatively affected and the cohesion in the cement paste-aggregate interface transition zone decreased.

- It is thought that the presence of C-A-S-H gels is important in the microstructure findings of the samples with waste glass additive.

In future studies, we think that detailed investigation of high temperature behavior or thermal properties in the production of cement-based building materials with waste glass additives is important in terms of recycling of wastes and ecological efficiency.

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