

DEVELOPMENT OF REFRACTORY CONCRETE USING GLASS GRINDING WASTE SAND

RAZVOJ V OGNJU ODPORNEGA BETONA Z UPORABO MLETEGA ODPADNEGA STEKLA

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This study investigates the utilization of glass grinding waste sand as an alternative fine aggregate in the formation of refractory concrete. Experimental refractory-concrete samples were subjected to sintering at 1200 °C, followed by a comprehensive evaluation of their physical and mechanical properties. The parameters assessed included flexural strength, volume density, shrinkage/expansion behavior upon drying and firing, thermal shock resistance, and thermal conductivity, following Vietnamese standards. Additionally, the mineral composition was determined using X-ray diffraction, while the microstructural characteristics were analyzed via scanning electron microscopy. Experimental findings indicate that incorporating 5 wt.% of glass grinding waste sand enhances the degree of sintering in refractory concrete at elevated temperatures while maintaining its essential refractory properties. The XRD analysis revealed the presence of α -Al₂O₃ and γ -Al₂O₃ phases, contributing to the refractory concrete's improved heat resistance and thermal shock resistance. The SEM analysis corroborated these findings by illustrating the microstructural modifications imparted by the glass grinding waste sand. Incorporating glass grinding waste sand at an optimal concentration of 5 w/% promotes sintering at high temperatures and preserves the critical refractory characteristics of concrete. This study underscores the potential of glass grinding waste sand as a viable alternative fine aggregate for refractory concrete, contributing to material sustainability and performance improvement.

Keywords: refractory concrete, corundum waste sand, glass grinding waste sand

V članku avtorji opisujejo študijo uporabnosti mletega odpadnega stekla kot alternativo finim agregatom za izdelavo v ognju odporne betona. Eksperimentalni vzorci refraktorskega betona so bili sintrani pri 1200 °C. Sledilo je ovrednotenje fizikalnih in mehanskih lastnosti izdelanih vzorcev oziroma preizkušancev. Parametri, ki so jih ocenjevali so bili: upogibna trdnost, volumska gostota in skrček oz. širjenje. V odvisnosti od načina sušenja in sintranja so ugotavljali tudi njihovo odpornost proti termičnemu šoku in toplotno prevodnost v skladu z lokalnimi standardi. Mineralno sestavo vzorcev so določili z rentgensko difrakcijo (XRD), med tem ko so mikrostrukturne značilnosti vzorcev betona analizirali s pomočjo vrstičnega elektronskega mikroskopa (SEM). Eksperimentalne ugotovitve so pokazale, da dodatek 5 mas.% mletega odpadnega stekla izboljša sinterabilnost refraktorskega betona pri visoki temperaturi pri čemer se ohranijo vse njegove lastnosti, ki so pomembne za uporabo pri povišanih temperaturah. XRD analize so pokazale prisotnost faz α -Al₂O₃ in γ -Al₂O₃, kar prispeva k njegovi izboljšani termični stabilnosti in odpornosti proti termičnim šokom. SEM analize pa so potrdile zgornje ugotovitve in mikrostrukturne spremembe zaradi prisotnosti mletega odpadnega stekla. Optimalna vsebnost mletega odpadnega stekla je 5 w/%. Ta izboljša sinterabilnost pri visokih temperaturah in kritične visoke temperaturne lastnosti betona. S pomočjo te študije so avtorji pokazali možnost uporabe mletega odpadnega stekla, kot zamenjavo za fini agregat v proizvodnji ognje varnega betona. Dodatek prispeva tudi k izboljšanju trajnosti in drugih njegovih za izdelavo in uporabo pomembnih lastnosti.

Povzetek: ognje varni beton, pesek iz odpadnega korunda in mletega odpadnega stekla

1 INTRODUCTION

In alignment with the nation's trend toward industrialization and modernization, the demand for refractory materials has experienced a significant increase in recent years. Refractory materials, particularly those used as construction materials for industrial furnaces operating at elevated temperatures, have become increasingly essential. Among these materials, refractory concrete is a crucial product for constructing kilns. Refractory concrete comprises a mixture of aggregates and binders, which can solidify in ambient air or under high-temperature conditions. It is commonly utilized for the construction

and binding of refractory bricks.¹ Additionally, refractory concrete is employed to fill refractory linings and seams, ensuring structural integrity and thermal stability. The formulation of refractory concrete involves four primary components: aggregate, plasticizer, binder, and water.² The specific composition involving these components is meticulously designed to ensure compatibility with the properties of the refractory bricks. The aggregates provide the structural framework, the plasticizer enhances workability, the binder facilitates the setting and hardening process, and water is the medium for mixing and hydration.³ A precise formulation and composition are critical for achieving desired performance characteristics, including high-temperature resistance, thermal shock resistance, and mechanical strength.

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In the 21st century, ecological issues and environmental protection are crucial worldwide. Human activities have rapidly reduced the available natural resources and created large wastes or by-products. Most of them cannot be recycled directly. The use of waste materials in producing new materials is an emerging research direction attracting increasing attention and development. In the field of construction, studies are progressively demonstrating that geopolymers materials have the potential to replace conventional cement products. Geopolymers, which serve as binding agents, can be synthesized from waste materials.⁴ Additionally, they exhibit superior heat resistance compared to traditional cement, making them suitable for fire-resistant structures.⁵

In line with this trend, construction materials for high-temperature applications are increasingly replacing traditional raw materials with waste-derived alternatives. Reusing and using waste sources as raw materials for producing refractory materials is also interesting. Therefore, improving the refractory concrete production technology and developing new heat-resistant products using secondary and waste materials without reducing durability and quality are currently very interesting.⁶ N. H. Thang et al. have demonstrated that waste materials such as ceramic fiber waste, diatomaceous earth, and rice husk ash can serve as alternative raw materials, producing refractory materials capable of withstanding temperatures exceeding 1400 °C.^{7,8}

The use of new refractory materials and the waste generated from producing these materials is expanding in the metallurgical industry. These can be used as refractory materials for gutter linings in the blast furnace industry. This industry often uses coal tar, a secondary hazardous substance that contains components harmful to the ecosystem, which has just been replaced by substances capable of ecological cleaning.⁹ F. R. Pereira et al. studied using four wastes, including Al-rich anodic sludge, sludge from the water purification/purification process, casting sand, and mud from the natural granite cutting process to manufacture refractory concrete.¹⁰ D. L. Giordani et al. also studied using thermoelectric ash and industrial ash as raw materials to produce refractory concrete. The results showed that adding ash significantly increased the melting point regardless of the type. In addition, concrete using thermoelectric ash would have fewer cracks in the final product.^{11,12} S. Fomenco et al. produced refractory bricks through the co-combustion of metallurgical waste. The resulting products exhibited

a thermal conductivity of 0.51–1.02 W/mK and compressive strength of 2.7–15.8 MPa.¹³

In this study, glass grinding waste sand was used to replace fine aggregates in the production of refractory concrete. Glass grinding waste sand is a type of waste generated later in the glass grinding process. Its ingredients include corundum and glass powder. The main chemical composition is Al₂O₃ and SiO₂, which can be replaced entirely as aggregates in the production of refractory concrete. Using waste sand as a raw material for producing refractory concrete helps to proactively source raw materials in our country's refractory concrete industry while simultaneously addressing waste disposal in the glass industry. However, an excessive amount of glass may compromise the refractory properties of a product. Therefore, it is crucial to carefully assess the optimal replacement content to ensure that the material's performance, particularly its thermal resistance and structural integrity, is not adversely affected. The feasibility of using glass grinding waste sand as a substitute thus depends on finding the balance between the desired refractory characteristics and sustainability. This consideration is essential for determining the appropriate glass-to-chamotte-sand ratio in production.

2 EXPERIMENTAL WORK

2.1. Materials

Glass grinding waste sand (GGWS) was taken from the Viglacera float glass factory. The chemical composition of GGWS is presented in **Table 1**. The mineral com-

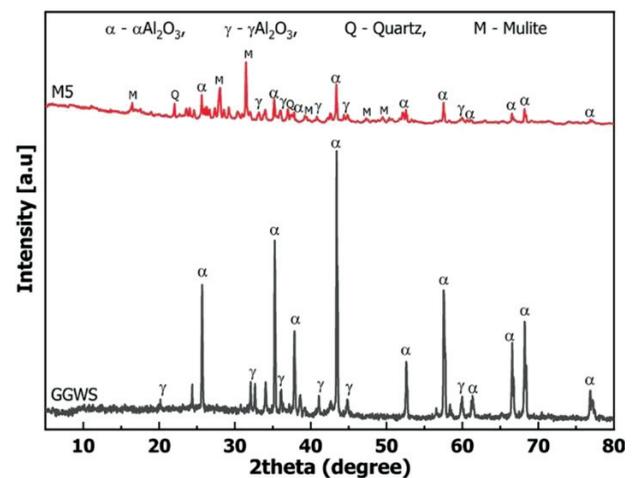


Figure 1: XRD patterns of GGWS and M5 sample

Table 1: Chemical compositions of the raw materials (w/%)

Raw materials	Al ₂ O ₃	SiO ₂	TiO ₂	Fe ₂ O ₃	NiO	R ₂ O	RO	MKN	Others
GGWS	39.20	48.80	6.40	1.02	1.57	0.20	0.26	0.15	2.40
RC	53.52	7.25	–	1.15	–	0.28	–	–	2.54
CC & FC	57.14	38.3	2.2	1.9	–	0.65	0.29	0.11	0.22
AC	5.23	78.29	–	0.25	–	1.15	2.36	10.59	2.13

position of GGWS includes α -Al₂O₃ and γ -Al₂O₃ (**Figure 1**).^{14,15} From the chemical and mineral composition of GGWS, it is clear that the main components are highly refractory minerals (α -Al₂O₃, γ -Al₂O₃), so it can be expected that this raw material can be used in the construction of refractory concrete. In this context, γ -Al₂O₃ represents the low-temperature allotropic form of Al₂O₃, while α -Al₂O₃ corresponds to the high-temperature allotropic form.

Besides GGWS, refractory concrete also uses other materials such as refractory cement (RC), coarse chamotte (CC), fine chamotte (FC), and active mineral additives (AC). The chemical compositions of these materials are shown in **Table 1**. RC functions as the binder, strengthening refractory concrete through its hydration with water. CC serves as the coarse aggregate, forming the structural framework of the concrete. Additionally, the large particle size of CC enhances the thermal shock resistance of the refractory concrete. FC acts as the fine aggregate, filling the voids of the CC particles. As a result, the sintering ability of the refractory concrete improves when exposed to high temperatures. AC is an active additive that enhances the post-hydration strength of the concrete.

2.2. Methods

GGWS was mixed with the other materials, including RC, CC, FC, and AC (**Figure 2**). The ingredients were mixed according to the composition shown in **Table 2**.

Table 2: Material ingredients

Samples	Raw materials (w/%)				
	RC	CC	FC	GGWS	AC
M0	25	40	40	0	5
M5	25	40	35	5	5
M10	25	40	30	10	5

The ingredients were mixed in different proportions in a planetary machine for 5 min. After mixing, the mixture was placed in a silicon mold. The mold was placed on a vibrator for 10 min to remove air bubbles. The mold



Figure 2: Raw materials: a) RC, b) CC, c) FC, d) GGWS, and e) AC

was then stored in a curing tank with a moisture content of not less than 90 % for 24 h. After that, the sample was removed from the mold and kept under stable curing conditions for 24 h. The sample was then dried at 110 °C for 24 h until completely dry. Finally, the dried sample was sintered at 1200 °C.

The bending strength was determined in accordance with the TCVN 6016:2011 standard. The volumetric density was identified in accordance with the TCVN 6530-3:1999 standard. Test samples were obtained after sintering at 1200 °C. The sample size was (20 × 20 × 100) mm. The shrinkage/expansion after drying and sintering was determined in accordance with the TCVN 10685-6:2018 standard. Test samples were obtained after sintering at 1200 °C. The sample size was (20 × 20 × 100) mm. The results represented the average of five tests. The thermal shock resistance was determined in accordance with the TCVN 6530-7:2000 standard. The samples were cylindrical, with a diameter of 50 mm and a height of 75 mm. The thermal conductivity was determined in accordance with the TCVN 9030:2011 standard. The coefficient of thermal conductivity was calculated according to Equation (1). **Figure 3** shows the experimental diagram for determining the coefficient of thermal conductivity. The test samples were unburnt samples at 1200 °C. They had a sheet-like shape with dimensions of (300 × 150 × 20) mm.

$$\lambda = \frac{q \cdot \delta}{\Delta t} = \frac{U \cdot I \cdot \delta}{2F\Delta t} \quad (1)$$

Here, λ is the coefficient of thermal conductivity (W/m°C); q is the heat flow (W/m²); δ is the thickness of the sample (m); Δt is the temperature difference between the inside and outside of the sample (°C); U is the voltage (V); I is the current (A); and F is the heat contact area of the sample (m²).

The phase compositions of samples were determined with X-ray diffraction (XRD). The X-ray used had CuK α radiation with a wavelength of 0.154 nm. The scanning range was from 5° to 80°, with a scanning step of 0.02°. The microstructure was observed with a scanning electron microscope (SEM). The scanning mode used was SE(M), with an applied voltage of 10 kV and a magnification of 200 times.

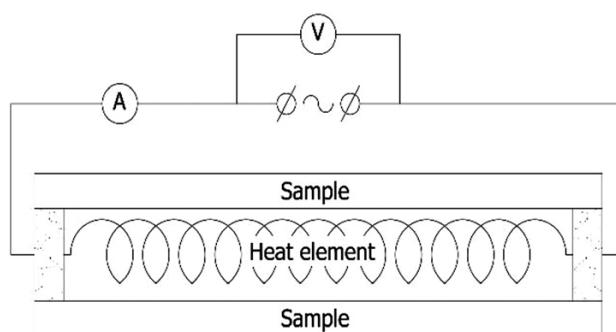


Figure 3: Experimental diagram for determining the thermal conductivity

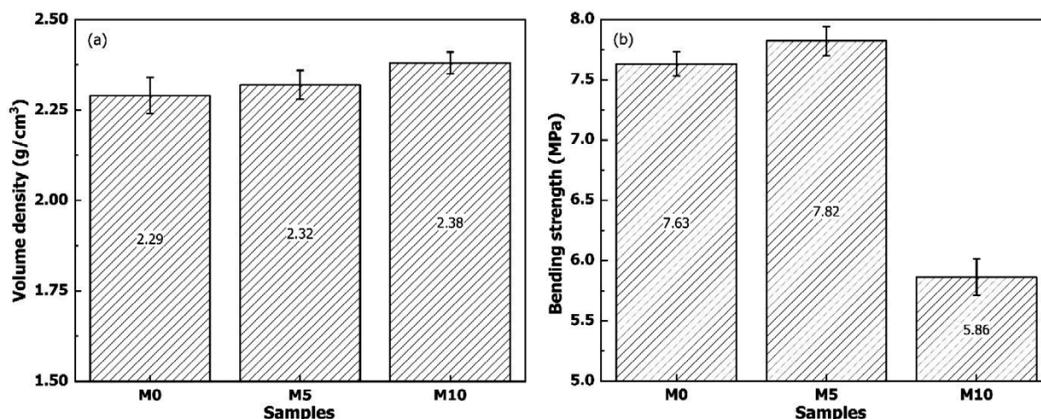


Figure 4: a) Volume density and b) bending strength of the samples

3 RESULTS AND DISCUSSIONS

After being shaped according to the composition in Table 1, the samples were sintered at 1200 °C for 4 h. After sintering at 1200 °C, samples were tested for mechanical and physical properties such as the bending strength and volumetric density. Figure 4 shows the results of determining the samples' volumetric density and bending strength. Each result is the average of five measurements.

The volume density results in Figure 4a show that when using GGWS in the aggregate composition, the volume density of the refractory concrete sample was almost unchanged. The highest sample volume density was 2.38 g/cm³ (M10 sample). The lowest sample volume density was 2.29 g/cm³ (M5 sample). The slight increase in the volume density of the samples using GGWS was due to the formed glass phase, which helped to fill in the pores. The lack of change in the volume density occurred when the input material changes could be explained based on the volume density of the wastes. The main component of GGWS is Al₂O₃. It has roughly the same volume density as FC in refractory concrete. Therefore, when replacing FC with GGWS, the volume density of the samples was almost unchanged. The results show that replacing FC with GGWS has little effect on the construction load.

Unlike the volume density, the strength of the samples changed when replacing FC with GGWS. Specifically, the strength of the sample with 5-% GGWS (M5 sample) increased. The strength of the sample with 10-% GGWS (M10 sample) strongly decreased. In the composition of GGWS, besides corundum, there is also waste glass resulting from a glass grinding process. Glass is an amorphous phase with a low melting point. It forms a liquid phase at high temperatures, creating favorable conditions for sintering. The increased strength of the M5 sample was due to the glass powder in GGWS. This glass powder created a liquid phase. The liquid phase created surface tension, allowing a rearrangement of the particles. As a result, the density of particles increased. In addition, the formed liquid phase also filled

in the pores among the particles. This was another reason why the strength of the refractory concrete increased after the sintering process. However, if the amount of waste sand was too large (M10 sample), the sample's mechanical strength was reduced. When too much GGWS was used, the amount of glass in the sample also increased. The liquid phase formed from glass also developed more easily at high temperatures. The low mechanical strength of the glass phase caused the sample to fail when the force was sufficiently increased. This was the reason for the decreased strength of sample M10.

Besides testing the physical and mechanical properties, the refractory concrete samples were also investigated for thermal properties such as shrinkage/expansion, shock temperature resistance, and thermal conductivity. Figure 5 shows the survey results for shrinkage/expansion upon drying and heating.

The results of shrinkage/expansion show that samples shrank when using GGWS. Upon drying shrinkage/expansion at 110 °C, when using GGWS, the samples shrank after drying. The volume change due to the polymorphic change of SiO₂ is the cause of this phenomenon.

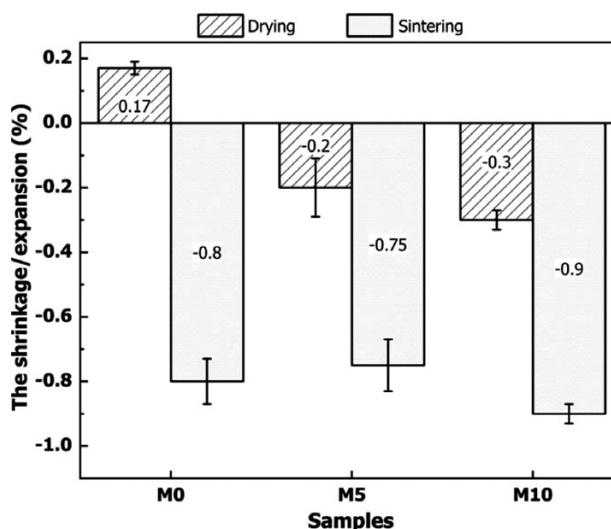


Figure 5: Shrinkage/expansion after drying and sintering

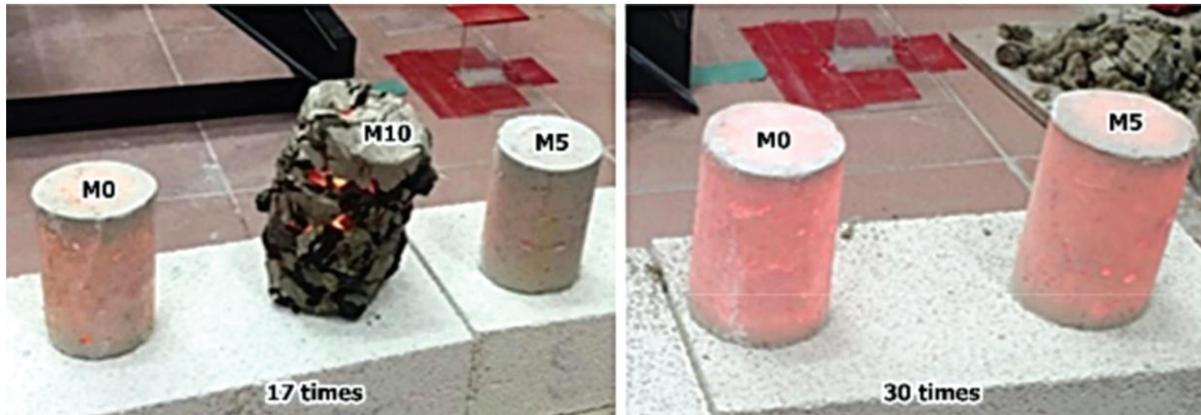


Figure 6: Thermal shock resistance results of the samples

As for the sample without waste sand, the sample expanded at 110 °C. At the sintering temperature of 1200 °C, all the samples, with and without GGWS, shrank after sintering. The high-temperature shrinkage, in this case, was due to the rearrangement of the particles during sintering, resulting in a higher density of the samples. However, the high-temperature shrinkage of the samples at 1200 °C was only from 0.75 % to 0.9 %. Therefore, the refractory concrete samples using GGWS still retained their volume stability at high temperatures, not affecting the structure of the building. Apart from the shrinkage/expansion behavior after drying and sintering, thermal shock resistance also contributes to the durability of refractory concrete during heating and cooling cycles. The thermal shock resistance was evaluated based on the TCVN 6530-7:2000 standard.

Figure 6 shows the results for the thermal shock resistance of the samples. It demonstrates that the M10 sample was destroyed after 17 heat shocks. The M0 and M5 samples withstood 30 heat shocks due to their thermal shock resistance. The results for the volumetric weight and strength showed that GGWS increases the sintering degree for refractory concrete. The sintering degree is affected by the glass phase formed from the glass in GGWS. However, in terms of thermal properties, glass is the component that reduces the thermal shock resistance. Glass has a low coefficient of thermal conductivity, which causes thermal stress when the temperature of the sample changes suddenly. Thus, when the amount of GGWS in the M10 sample was too high, the thermal shock resistance decreased. On the other hand, the amount of glass in the M5 sample was not too large, so its compaction increased, ensuring its thermal shock resistance. Refractory concrete that includes 5 w/% GGWS can be used for intermittent and continuous thermal equipment. The thermal shock resistance of refractory concrete is influenced by various factors, among which thermal conductivity is critical. High thermal conductivity allows the material to exhibit better thermal shock resistance. Consequently, the thermal conductivity of refractory concrete was also investigated.

Figure 7 shows the results of determining the thermal conductivity of the samples. The results show that the thermal conductivity increases with increasing temperature of the samples. Increasing thermal conductivity with temperature is a common phenomenon in ceramic materials.^{16,17} As the temperature rises, the heat flow density increases, so the material's thermal conductivity also increases. In all samples, the coefficient of thermal conductivity becomes unstable in a temperature range from 300 °C to 500 °C. This temperature range corresponds to the combustion of organic impurities and water dehydration in the minerals of refractory cement.^{18,19} These processes can be described with the chemical reactions from Equations 2–4. A change in the material's structure in this temperature range causes a substantial change in thermal conductivity.^{20,21} When the dehydration reaction is complete, the strength of the sample, provided by the cement, is lost. However, the test to determine the bending strength of the samples shown in **Figure 4b** still showed that the samples kept their mechanical strength. This result proves that the strength of formation at high temperatures is due to the material agglomeration process.

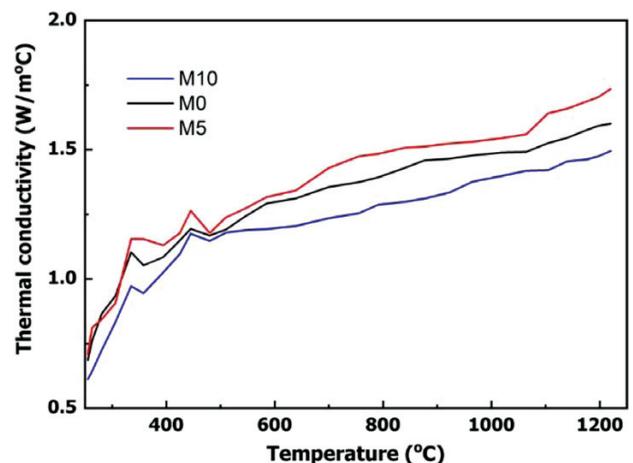


Figure 7: Thermal conductivity results for the samples

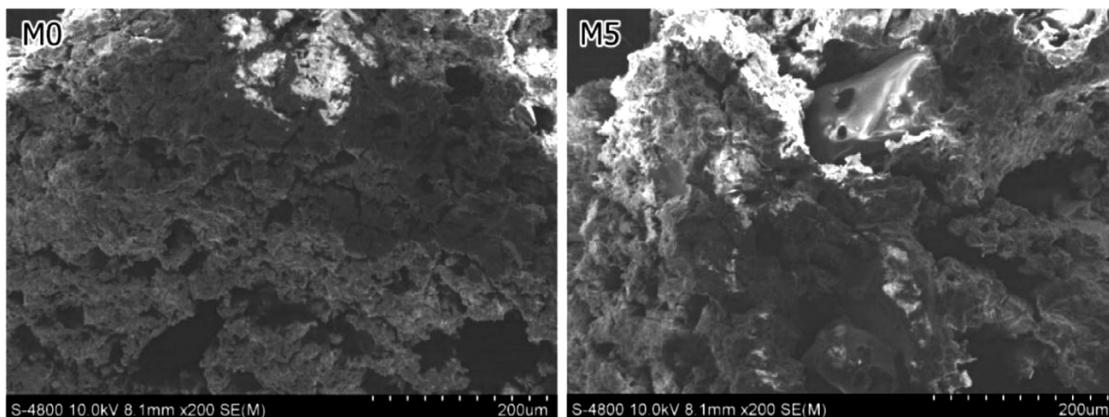


Figure 8: Microstructures of the M0 and M5 samples



(*)a, b, c, d, e, f, y, and z are the coefficients.

When comparing the coefficients of thermal conductivity between samples, the results from **Figure 7** show that the coefficient of thermal conductivity decreases when the GGWS content is too high (10 w%). The difference in the thermal conductivity of the samples is especially evident at temperatures above 500 °C. This is because the glass phase increases when the GGWS amount is too high. The glass phase has a low coefficient of thermal conductivity, so when its amount is too high, it reduces the thermal conductivity of refractory concrete. Due to a low coefficient of thermal conductivity, the temperature difference between different regions in the sample also increases with a temperature change. Therefore, the rate of shrinkage/expansion in other regions is also different. The samples exhibit internal boundary stress within these regions. As a result, the thermal shock resistance of refractory concrete decreases. Contrary to the M10 sample, M5 sample has a higher coefficient of thermal conductivity than sample M0. SEM images of the M0 and M5 samples show microstructure differences after 30 thermal shocks.

Figure 8 shows the microstructures of the M0 and M5 samples obtained with SEM after 30 thermal shock tests. The microstructure images show that the samples were still well bonded after the heat shock test. In addition, microcracks on the surfaces show that the sample using GGWS underwent less microcracking than the sample not using GGWS. The reduction of cracks was due to the GGWS containing Al_2O_3 minerals. Al_2O_3 minerals exhibit good thermal conductivity, increasing the material's thermal shock resistance. Al_2O_3 compensates for the reduction in the refractory properties caused by the glass in GGWS. The existence of Al_2O_3 minerals was proven with XRD.

The XRD patterns in **Figure 1** show that in the M5 sample the main mineral compositions are quartz (SiO_2), mullite ($3\text{Al}_2\text{O}_3 \cdot 2\text{SiO}_2$), $\alpha\text{-Al}_2\text{O}_3$, and $\gamma\text{-Al}_2\text{O}_3$. Quartz was present at diffraction positions of 20.83°, 26.62°,

and 39.44°. $\alpha\text{-Al}_2\text{O}_3$ was present at diffraction positions of 16.48°, 36.31°, 33.31°, 39.36°, 48.15°, and 49.67°. $\gamma\text{-Al}_2\text{O}_3$ was present at diffraction positions of 25.48°, 35.1°, 37.5°, 43.2°, 52.5°, 57.4°, 61.3°, 66.2°, 67.9°, and 76.9°. Quartz and mullite are components of CC and FC of concrete. $\alpha\text{-Al}_2\text{O}_3$ and $\gamma\text{-Al}_2\text{O}_3$ are the components of GGWS. These minerals have high fire resistance, so they make concrete fire resistant. In addition, as mentioned above, $\alpha\text{-Al}_2\text{O}_3$ and $\gamma\text{-Al}_2\text{O}_3$ with high thermal conductivity contribute to improved refractory properties.

The results of the analysis of mechanical and thermal properties show that the replacement of 5 % FC with GGWS was an appropriate choice for producing refractory concrete. It could increase the sintering degree of concrete at high temperatures but did not change the refractory properties. The results indicate that GGWS can be successfully used as a substitute for fine chamotte sand in the refractory mortar and concrete production. This substitution not only maintains but, in some cases, enhances the desired properties of final products, such as strength and durability. Additionally, this approach provides a sustainable solution for the disposal of GGWS, which is often an underutilized by-product. By incorporating this waste material, industries can reduce the environmental impact of glass waste disposal and the extraction of natural resources.

5 CONCLUSIONS

This study used glass grinding waste sand to replace FC of refractory concrete. The results showed that when replacing this fine aggregate with 5 w% GGWS the sintering degree of refractory concrete is higher than that of regular concrete. The concrete that used GGWS still ensured thermal shock resistance and volume stability at high temperatures. The analysis of the mineral composition and microstructure also showed that refractory concrete using GGWS included quartz, mullite, $\alpha\text{-Al}_2\text{O}_3$, and $\gamma\text{-Al}_2\text{O}_3$. Minerals $\alpha\text{-Al}_2\text{O}_3$ and $\gamma\text{-Al}_2\text{O}_3$ exhibit high

heat resistance and thermal conductivity, thus increasing the concrete's fire resistance and thermal shock resistance. Refractory concrete using GGWS can be used for constructing intermittent and continuous thermal equipment.

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