

Research Article

Research of Glass-Ceramic Material Synthesis Through Waste By-Product Sintering from Mining and Metallurgical Activities

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Abstract This study analyzes the possibility of producing glass-ceramics from chromite tailings, coal fly ash (Class F), and red mud industrial wastes in Türkiye. Unlike previous studies that typically used binary mixtures or different compositional ratios, this study introduces a novel ternary combination with a fixed 1:2:3 ratios (fly ash: chromite tailings: red mud). This specific ratio was optimized through experimental trials and found to enhance sinterability, mechanical strength, and microstructural stability. The glass-ceramics obtained using this ratio demonstrated compressive strength, water absorption, and density of 12.21 MPa, 0.44%, and 2.05 g/cm³, respectively. The resultant products were analyzed by XRD and leaching tests, which confirmed quartz-based crystalline phases and negligible leaching of toxic metal ions. These findings propose a new strategy for environmentally benign waste recycling, aligned with green chemistry principles and the UN Sustainable Development Goal 12.

Keywords: Sintering, Glass-ceramics, Refractories, Mining tailings

1. INTRODUCTION

The production of glass-ceramics from mining and metallurgical wastes as composite materials offers a promising pathway for both waste utilization and recycling. Such materials have potential applications across various industrial sectors, including factory flooring, heat-resistant utensils, and other engineering products. They comply with compositional, toxicity leaching, and processing standards, demonstrating their feasibility as ideal industrial products [1, 2]. The generation of solid wastes and the resulting environmental pollution from mining and metallurgical operations necessitate the development of robust waste management policies. In Türkiye, chromite mining alone produced approximately 6.5 million tons of solid tailings in 2021 [3]. Additionally, the Bayer process used for alumina production from bauxite generates red mud as a by-product—typically amounting to 1–1.5 times the mass of the alumina extracted. The Seydişehir (Konya, Türkiye) alumina refinery produces about 200,000 tons of alumina annually, accompanied by roughly 300,000 tons of solid red mud residues [2] [4]. Moreover, over 50 coal-fired power plants in Türkiye generate more than 13 million tons of fly ash each year (both Class C and Class F). Despite this, the utilization rate of fly ash remains considerably lower than that of developed countries, particularly in applications such as building materials and concrete production [5-7]. In response to this need, numerous national and international studies have been conducted to enhance the economic value of fly ash while simultaneously mitigating its environmental impact.

In this research, solid wastes from chromite mining, red mud, and fly ash were used to produce glass-ceramic building materials through the sintering method, with quartzite, china clay, and sodium carbonate serving as additives. Previous studies [8-27] have explored various industrial waste sources for their potential in glass-ceramic production, primarily focusing on waste utilization and recycling. Most of these earlier works [9, 12, 13, 15, 22, 28] investigated binary mixtures—such as red mud–chromite tailings or fly ash–industrial waste—at different compositional ratios. However, no comprehensive study has yet examined the specific ternary mixture (fly ash: chromite tailings: red mud = 1: 2: 3) employed in this research. The synergistic combination of these three waste materials in this ratio was found to enhance sinterability, mechanical strength, and heavy metal immobilization efficiency. This experimental study provides a valuable technical reference for the simultaneous disposal and valorization of mining and metallurgical wastes through glass-ceramic production.

2. MATERIALS

Chromite tailings were obtained from a local chromite mining company (Akmetal, Adana, Türkiye). Red mud was supplied by Eti Alüminyum Corp. Fly ash (Class F, as defined in ASTM C618) was collected from the Sugözü Thermal Power Plant (Türkiye). Kaolin was provided by Zafer Maden (Balıkesir, Türkiye), and quartzite was supplied by a local supplier. Poly(vinyl alcohol) (PVOH, PVA, or PVAI), a water-soluble synthetic polymer with the general formula $[\text{CH}_2\text{CH}(\text{OH})]_n$, was used as a binding agent for all experiments. It was supplied by Sigma-Aldrich (Product No. 341584; CAS No. 9002-89-5). Sodium carbonate (Na_2CO_3) of analytical grade (CAS No. 497-19-8) was also used as an additive and obtained from a local supplier.

3. METHODS

Mineralogical analysis of the raw materials was conducted using a PANalytical EMPYREAN powder X-ray diffractometer (Cu $K\alpha$ radiation, 20–50 kV, 5–60 mA) with a scanning range of $2\theta = 5\text{--}70^\circ$ and a step rate of $2^\circ/\text{min}$. Phase identification was performed using *HighScore Plus* software with the COD 2020 database. Particle size distribution was determined by the dry mechanical sieving method for particles coarser than $75\ \mu\text{m}$, while a *Malvern Mastersizer 3000* instrument was used for sub-sieve fractions ($<75\ \mu\text{m}$). Elemental (oxide) compositions of the wastes and additives were analyzed using a *PANalytical MiniPal* X-ray fluorescence (XRF) spectrometer following the standard DIN 51001 [29]. For verification, hydrofluoric acid digestion followed by atomic absorption spectrophotometry (AAS) was also applied. Loss on ignition (LOI) was determined by heating pre-weighed dry samples (dried at $105\ ^\circ\text{C}$) at $1000\ ^\circ\text{C}$ for 2 h, followed by cooling in a desiccator. Table 1 presents the chemical compositions and sources of the solid wastes and additives used in glass-ceramic production.

Table 1: Chemical analysis of the waste samples and additives used in the batch preparation.

Oxide, wt. %	Chromite waste (tailings)	Red mud	Fly Ash (Class F)	Quartzite	Kaolin
SiO_2	43.00	15.46	46.00	98.47	71.67
Al_2O_3	-	18.87	23.00	0.40	18.25
Fe_2O_3	37.30	35.56	9.21	0.30	0.30
TiO_2	-	5.82	2.10	0.04	0.36
CaO	3.66	2.06	14.20	0.01	0.13
MgO	-	0.16	-	0.02	0.09
Na_2O	-	11.32	-	0.03	-
K_2O	-	0.38	1.60	0.14	0.49
MnO	-	-	0.09	-	-
SO_3	-	0.32	1.60	-	1.00
Cr_2O_3	2.06	-	0.03	-	-
NiO	3.06	-	0.01	-	-
LOI at $1,000\ ^\circ\text{C}$	7.32	7.96	5.37	0.56	7.66

A sintering temperature range of $1000\text{--}1200\ ^\circ\text{C}$ was selected based on preliminary experiments and literature data, as this range is suitable for initiating crystallization in alumino-silicate-rich waste-derived glass-ceramic systems. This temperature window provides sufficient viscosity reduction for particle bonding while preventing excessive melting or deformation. A dwell time of 1 h was adopted to promote adequate phase transformation and crystal growth, essential for enhancing mechanical and physical properties. The raw materials were dry-ground in a ball mill for 30 min to achieve a particle size of $d_{100} = 100\ \mu\text{m}$ under closed-loop conditions. The batch components were then homogenized using a laboratory mixer for 10 min. Each batch ($75 \pm 0.1\ \text{g}$) was prepared for the glass-ceramic test compositions. The binder (PVA) was dissolved in distilled water at $85\ ^\circ\text{C}$ for 1 h and then mixed with the batch materials. The mixture was dried in an oven at $75\ ^\circ\text{C}$ for 3 h. The dried powder was molded in a $15\ \text{cm}^3$ oiled cylindrical steel mold and pressed for 2 min under $200\ \text{kg}/\text{cm}^2$ using a 30 T laboratory hydraulic press. Sintering of the green compacts was performed in an electrically heated muffle furnace between 1000 and $1200\ ^\circ\text{C}$ for 2 h at a heating rate of $5\ ^\circ\text{C}/\text{min}$. Cooling occurred naturally within the furnace to room temperature. All sintering tests were repeated twice, producing four specimens for each experimental condition.

The apparent solid density (ρ_a) of the sintered specimens was determined according to ISO 18754:2020(E), Method A1 (liquid displacement under boiling conditions). The test piece was impregnated by maintaining it in a liquid under boiling conditions. For the calculation of the apparent solid density of the glass-ceramic [30, 31], an equation given below was applied:

$$\rho_a = \frac{m_1}{m_1 - m_2} \times \rho_1 \quad (1)$$

where

ρ_a is the apparent solid density, kg/m^3 ;

m_1 is the mass of the dry test specimen, kg;

m_2 is the apparent mass of the immersed test specimen, kg;

ρ_1 is the density of the immersion liquid at the temperature of the test, kg/m^3 .

Water absorption was determined following EN 13755:2008 [32]. Dried specimens were weighed to a constant mass (m_d), immersed in water at atmospheric pressure for 48 h, then wiped with a damp cloth, and reweighed (m_s). Measurements were repeated every 24 h until a constant saturated mass was achieved. Water absorption (A_b) was calculated using Equation (2):

$$A_b = \frac{m_s - m_d}{m_d} \times 100 \quad (2)$$

where

m_d is the mass of the dry specimen, g;

m_s is the mass of the saturated specimen,

g; A_b is the water absorption at atmospheric pressure, %.

The compressive strength of the glass-ceramic specimens was evaluated using the Point Load Testing method in accordance with the International Society for Rock Mechanics (ISRM) standard [33]. The testing apparatus consisted of a point load frame with a pressure-measuring unit (Figure 1). The point load strength index (Is_{50}) was calculated using Equation (3):

$$Is_{50} = \frac{P}{D_e^2} \quad (3)$$

where

P is the failure load (N).

D_e is the equivalent core diameter (cm).

For each composition, four specimens were tested, and the average Is_{50} value was taken as the compressive strength of the material.

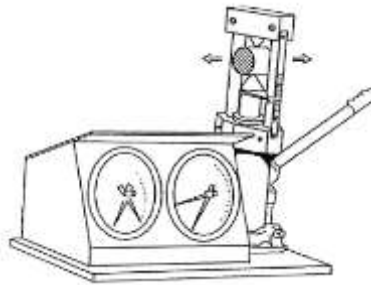


Figure 1: The point load tester.

3.1. Leaching of the Glass-Ceramics

The potential leachability of toxic substances from the produced glass-ceramics was evaluated following the Toxicity Characteristic Leaching Procedure (TCLP, Method 1311) [34]. Sintered glass-ceramic specimens were first ground to a particle size of <9.5 mm and then leached using an acetic acid solution adjusted to $\text{pH } 2.88 \pm 0.5$, maintaining a liquid-to-solid ratio of 20:1 mL/g. The resulting leachates were filtered through $0.45 \mu\text{m}$ membrane filters and acidified with nitric acid to $\text{pH } 2$ prior to analysis. The concentrations of heavy metals in the leachates were determined using a PerkinElmer 900 H Atomic Absorption Spectrometer (AAS).

4. RESULTS AND DISCUSSION

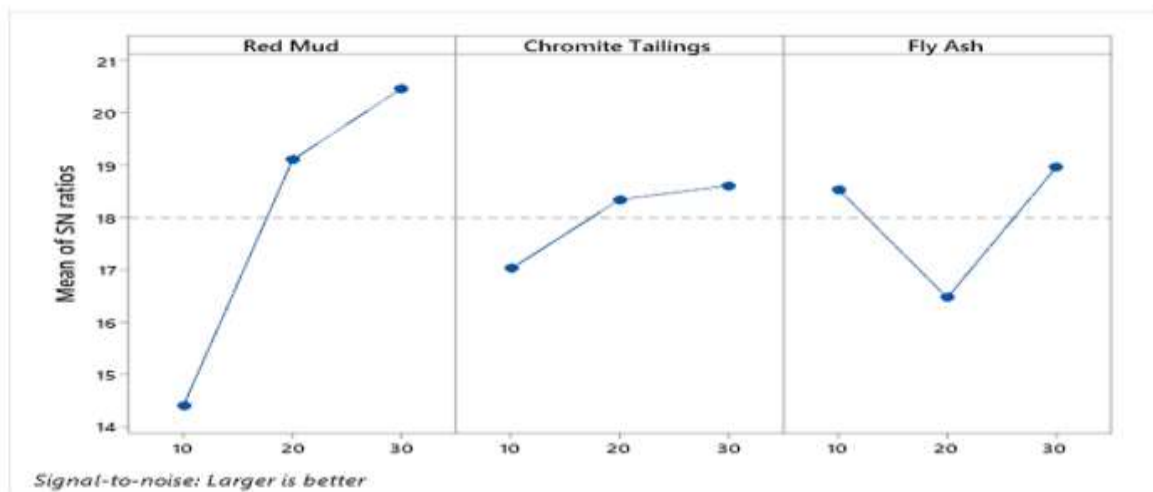
4.1. Raw Materials

Silica and iron were the dominant components in the chromite tailings, while red mud was primarily composed of silica, iron, and aluminum oxides. Fly ash mainly contained silica and alumina (Table 1), consistent with the results of the XRD analyses. As shown in Figure 2a, the principal crystalline phases identified in the chromite waste were lizardite ($\text{Mg}_3(\text{Si}_2\text{O}_5)(\text{OH})_4$), fayalite (Fe_2SiO_4), olivine ($(\text{Mg,Fe})_2\text{SiO}_4$), and tridymite, a high-temperature polymorph of silica (SiO_2). The XRD pattern of red mud (Figure 2b) revealed quartz (SiO_2), ferrite spinel ($(\text{Mg,Fe})(\text{Fe,Al})_2\text{O}_4$), mullite ($\text{Al}_6\text{Si}_2\text{O}_{13}$), and hematite (Fe_2O_3) as the major phases. The F-class fly ash (Figure 2c) mainly contained quartz, mullite, and calcite. Among the additives, quartzite (Figure 3a) consisted of quartz, periclase (MgO), rutile (TiO_2), and muscovite ($\text{KAl}_2(\text{AlSi}_3\text{O}_{10})(\text{F,OH})_2$), while kaolin (Figure 3b) contained kaolinite ($\text{Al}_2\text{Si}_2\text{O}_5(\text{OH})_4$) and cristobalite (SiO_2), the latter being a high-temperature polymorph of silica formed above $1,400^\circ\text{C}$.

Table 2: Production conditions for producing glass-ceramics and their levels (wt.%).

Parameters	Symbol	Level 1	Level 2	Level 3
Red Mud	A	10	20	30
Chromite tailings	B	10	20	30
Fly ash	C	10	20	30

The full factorial design with an orthogonal array of Taguchi L9 (3³) for the experimental conditions needed for obtaining glass-ceramics is given in Table 3. It was aimed to obtain the product with higher point load index values. Therefore, the “larger is better” quality characteristic was used.



Level	Red Mud	Chromite Tailings	Fly ash
1	14,40	17,02	18,54
2	19,10	18,35	16,46
3	20,47	18,61	18,97
Delta	6,07	1,59	2,50
Rank	1	3	2

Figure 4: S/N values of the experiments.

For preliminary trials, green compacts were sintered at 1,200 °C for 2 h and annealed at 650 °C for 1 h [11, 28]. Temperature was identified as the key factor governing crystallization behavior and material properties. The resulting glass-ceramic samples exhibited reddish-black coloration due to the presence of iron and titanium oxides. Kaolin was kept constant in subsequent experiments as a dual source of Al₂O₃ and SiO₂, enhancing melt viscosity and structural stability at elevated temperatures. Further sintering tests (Table 5) were conducted to optimize the temperature conditions. The best-performing glass-ceramic produced from the 1:2:3 mixture displayed an apparent solid density of 2.05 g/cm³, average water absorption of 0.44%, and point load index of 12.21 MPa. XRD results (Figure 5) indicated that the dominant crystalline phases were quartz (SiO₂), rutile (TiO₂), and kaolinite, consistent with the mineral constituents of the raw materials. Microstructural examination using SEM (QUANTA FEG 650) revealed a vitreous, amorphous matrix with embedded crystalline phases and isolated pores (Figure 6). This microstructure contributed to the material’s high density and compressive strength. For various pyro-metallurgical and refractory applications, particularly those based on spinel and mullite phases, understanding phase equilibria in the Al₂O₃–FeO–Fe₂O₃–SiO₂ system is crucial. Relevant studies in the literature have provided extensive experimental and thermodynamic modeling data for this system [28].

4.3. Leaching Behaviors of Cr After Thermal Processes

The concentrations of leached elements from the sintered glass-ceramic products are presented in Table 6. The results demonstrated that the heavy metal concentrations in the leachates were well below the regulatory limits specified by TCLP standards. Consequently, the glass-ceramic products derived from mining and metallurgical wastes can be safely utilized for household and construction applications, such as ceramic tiles [35, 36].

Table 3: Full factorial design with an orthogonal array of Taguchi L9 (3³).








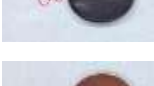

Experiment no	Red Mud (wt.%)	Chromite tailings (wt.%)	Fly ash (wt.%)	Point load index (MPa) (mean values)	Visual appearance of prepared glass ceramics
1	10	10	10	4.69 ± 0.07	
2	10	20	20	4.69 ± 0.05	
3	10	30	30	6.57 ± 0.07	
4	20	10	20	7.05 ± 0.06	
5	20	20	30	9.86 ± 0.09	
6	20	30	10	10.56 ± 0.09	
7	30	10	30	10.80 ± 0.1	
8	30	20	10	12.21 ± 0.1	
9	30	30	10	8.92 ± 0.07	

Table 4: ANOVA result of the experiments.

Source	DF	Adj SS	Adj MS	F-Value	P-Value
Red Mud	2	45.329	22.6646	33.24	0.029
Chromite Tailings	2	3.404	1.7018	2.50	0.286
Fly Ash	2	9.940	4.9699	7.29	0.121
Error	2	1.364	0.6819		
Total	8	60.036			

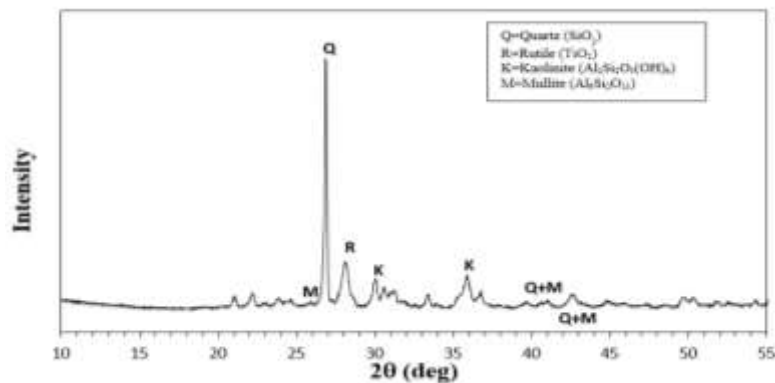







Figure 5. Diffractograms of the glass-ceramic heat treated at 1,200 °C.

Table 5: Mix design of the prepared glass-ceramics.

	Reference				
	A1	B1	C1	D1	E1
Chromite waste (g)	20	20	20	20	20
Red mud (g)	30	30	30	30	30
Fly Ash (Class F) (g)	10	10	10	10	10
Quartzite (g)	20	20	20	20	20
Kaolin (g)	20	20	20	20	20
Sodium carbonate (g)	1.5	1.5	1.5	1.5	1.5
Binding chemical (g)	2	2	2	2	2
Firing temperature (°C)	1,000	1,100	1,200*	1,200**	1,200***
Point load index (MPa)	0.68	1.14	9.68	10.80	12.21
Visual appearance of prepared glass ceramics					

* dwell time: 2 h

** dwell time: 30 min

*** no dwell time

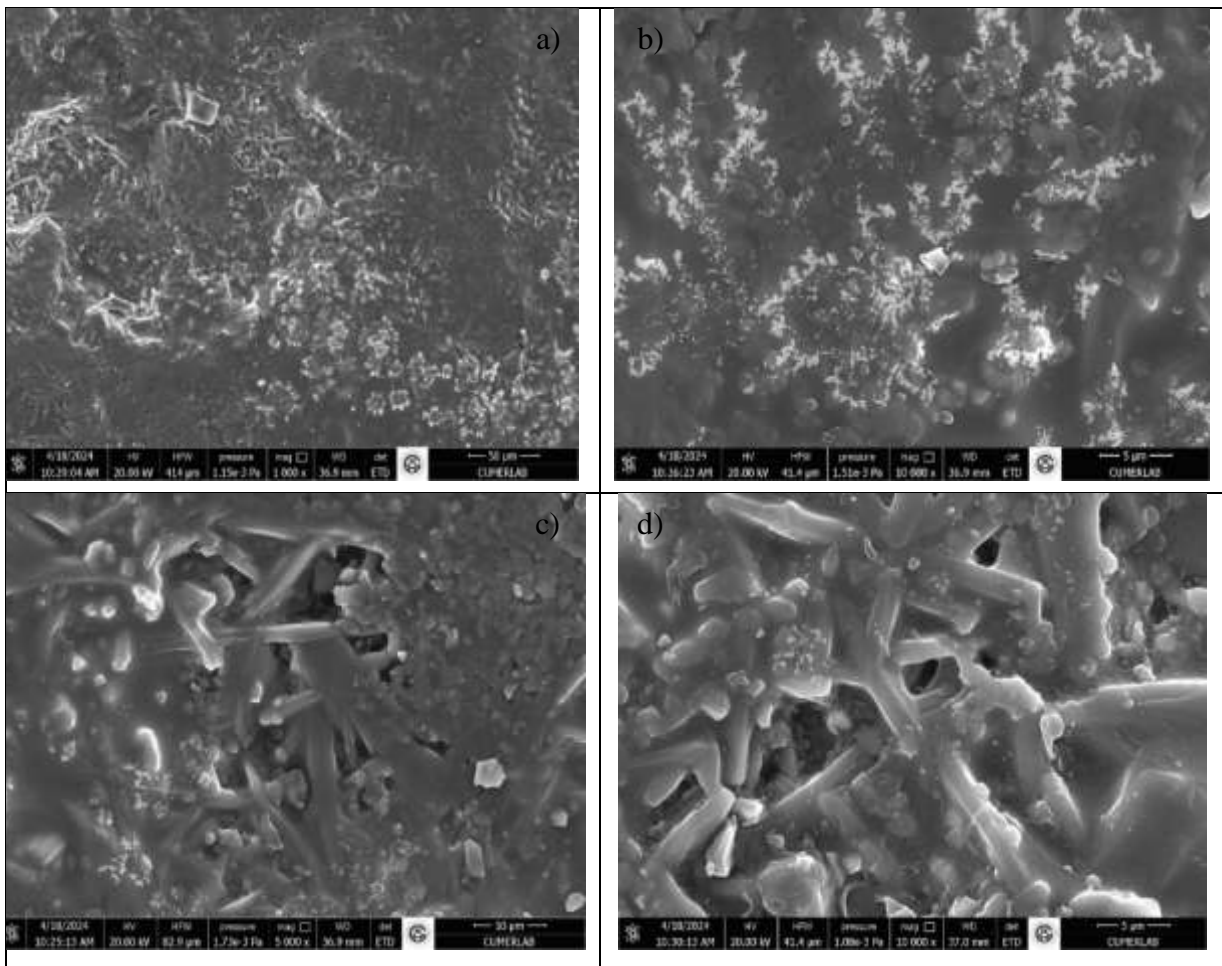


Figure 6: The microstructure of the glass-ceramic heat-treated at 1,200 °C at different magnifications: (c) aggregates of needle-like mullite crystals growing from the surface toward the center of the glass particles, and (c) interlocked mullite crystals.

Table 6: Chemical analysis of the leachate from glass-ceramic product applying TCLP-Method 1311.

Element	The leachate (mg/L)
Mn	0.14
Al	nd (<1 mg/L)
Fe	0.061
Cr-total	nd (<0.1 mg/L)
Cu	0.107
Ti	nd (<1 mg/L)
Ni	nd (<0.1 mg/L)
Pb	nd (<0.1 mg/L)
Ca	4.40

4.4. Potential Applications

In Türkiye, the production of environmentally friendly glass-ceramics is increasingly encouraged due to growing social and environmental awareness regarding energy consumption, waste accumulation, and depletion of natural resources. The valorization of industrial wastes such as red mud, chromite tailings, and fly ash offers a sustainable and low-cost pathway toward circular economy goals. Table 7 presents a comparative overview of the general properties of glass-ceramics for architectural applications, alongside similar materials reported in the literature [37-41].

In this study, only collection and grinding of the wastes to the required particle size were necessary, resulting in minimal production cost. However, the overall economic feasibility depends primarily on labor, waste transportation, energy consumption, operating costs, and governmental incentives. Therefore, further quantitative techno-economic and environmental assessments are recommended. The findings of this study suggest that waste-derived glass-ceramics provide an innovative, cost-effective, and environmentally sustainable waste management solution that promotes resource recovery and pollution mitigation.

Table 7: Comparison of the physical and technological properties of building glass-ceramics along with the commercial products and the material produced in this study.

	Glass-ceramic [37]		Commercial Glass-ceramics (typical ranges) [41].	Produced Material (This Study)
	Conventional	Neoparis		
Density, g/cm ³	2.4-3.6	2.7	2.4-2.8	2.05
Compressive strength, (MPa)	n.a.	12-56	>1000 MPa	12.21 (Point load index)
Water absorption, %	1-5	<1	<0.5	0.44
Porosity, %	-	-	<1-2%	n.a.
Hardness (Mohs)	-	-	6-7	n.a.
Thermal expansion (*10 ⁻⁶ K ⁻¹)	-	-	5-9	n.a.
Thermal shock resistance	-	-	High (better than glass, lower than ceramics)	n.a.
Acid/alkali resistance	-	-	Excellent	Excellent
Aesthetic properties	-	-	Glossy/matte, colorable, decorative designs	Glossy/matte, colorable, decorative designs

5. CONCLUSION

This study demonstrated the feasibility of producing glass-ceramics from fly ash, chromite tailings, and red mud with an optimal 1:2:3 ratio. The resulting materials exhibited favorable physical and mechanical properties, including high density and strength, and showed no significant leaching of heavy metals. Although the study was limited by the lack of detailed microstructural and thermal analyses, limited replicates of mechanical tests, and absence of economic and environmental evaluations, it nonetheless provides a valuable foundation for future research. The results highlight the potential of integrating industrial waste valorization with sustainable material design, offering a promising approach for regions affected by extensive mining and metallurgical activities.

Authors' Contributions

Oktay Bayat was primarily responsible for manuscript writing and data analysis, and contributed through critical review, editing, and overall supervision of the study. Mahmut Altiner and Muhammed Aboelgamel were responsible for specimen preparation, execution of experimental tests, and participation in data analysis.

Declaration of competing interest

No known competing financial interests or personal relationships could have appeared to influence the study reported in this paper was declared by the authors.

Data availability

Data will be made available on request.

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