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EFFECT OF ALUMINA ADDITION ON THE PROPERTIES OF GLASS-CERAMICS DERIVED FROM WASTE WINDOW GLASS VIA SINTERCRYSTALLIZATION

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Abstract

This study investigates the development of glass-ceramics from waste window glass using the sinter-crystallization technique. Waste glass was ground, sieved through a 100 µm mesh, and analysed by the X-ray fluorescence (XRF), which revealed a soda-lime-silica composition with SiO2 (59.80 wt%) as the dominant oxide, followed by Na2O (23.03 wt%) and CaO (10.09 wt%). Compacts were prepared with alumina additions ranging from 0 to 8 wt% in 2 wt% increments, pressed at 15 kN, air-dried for 24 hours, and sintered at 900°C with a heating rate of 5°C/min and a 1-hour hold to promote the formation of crystalline phases within the residual glass matrix. The X-ray Diffraction (XRD) analysis revealed wollastonite, quartz, and albite as the main crystalline phases. Increasing alumina content promoted wollastonite crystallization, which improved mechanical strength and chemical durability. For example, GC-1 (0 wt% Al2O3) contained 72.5 wt% wollastonite and 25.3 wt% quartz, while GC-3 (4 wt% Al2O3) exhibited higher crystallinity with 80.5 wt% wollastonite and 17.5 wt% quartz. The sample with the highest alumina content, GC-5 (8 wt% Al2O3), demonstrated superior hardness and resistance to sulfuric acid. These results confirm that alumina enhances the crystallization behavior of waste glass, enabling the production of durable glass-ceramic materials suitable for tiling applications.

Keywords
Acid resistance, crystallinity, hardness, phase transformation, sintering temperature, waste recycling

1. INTRODUCTION

Materials play a crucial role in science, technology, industry, and daily life (Guo *et al*.,2024). Glass is one widely used material with many applications. Each year, millions of tons of glass waste are produced globally, contributing to growing landfills of non-biodegradable materials (Fu *et al*., 2021). This not only harms the environment but also wastes valuable resources that could support technological and industrial progress. The overall global recycling of waste glass was very low, for example, in 2018, worldwide production of glass was about 130 million tons but only 21% of glass was subjected to recycling. Waste glass includes discarded items such as broken bottles, jars, and other glass pieces. These materials are non-biodegradable and pose environmental challenges in landfills. A large portion of glass waste can be collected and remelted to produce new glass products. Completely eliminating waste glass, or cullet, from glass-making industries worldwide is unrealistic. Therefore, recycling and reusing glass waste is essential for efficient resource management (Fu *et al*., 2021). Glass making is an energy-intensive process with costs on energy taking up 15% of total glass manufacturing costs. Although glass is recyclable, only 21% of global glass volumes is recycled per year (Harder, 2024). Recycling glass waste is not only environmentally necessary but also economically beneficial and viable as it reduces landfills and incinerators waste, conserve resources, saves energy because it can be remelted at a lower temperature, reduces environmental pollution, create jobs opportunity and stimulate local economy as well as protect natural habitats and landscapes among others (Wilburn *et al*., 2021).

There is a need to explore sustainable approaches to glass production using waste window glass, a non-biodegradable material with significant environmental impacts. One promising method is recycling waste to produce glass-ceramics, a versatile material with many applications. The glass-ceramics production process can use a wide range of waste materials, especially silicate-based wastes. These include waste glass (Aliyu and Salisu,2023), corn cob ash (Jaehong *et al*., 2015), rice husk ash (Aliyu and Salisu,2023), sugarcane bagasse ash (Norsuraya *et al*., 2016), fly ash (Rawlings *et al*., 2006) and blast furnace slag (Mihailova *et al*., 2011), among others.

Over the past 50 years, glassy materials have seen significant technological and industrial developments, surpassing many common materials such as ceramics (Richet *et al*., 2021). The concept of transforming glass into a crystalline material through controlled crystallization was first attempted by the French chemist Reaumur in the early 17th century. He heat-treated a glass bottle in sand and gypsum, aiming to produce a porcelain vase (Richet *et al*., 2021). In 1953, American inventor S.D. Stookey achieved a breakthrough with controlled crystallization of certain glasses, which expanded the theory of phase separation (Chakrabarti *et al*., 2022). Advances in glass manufacturing, from early core-forming methods to modern moulding and pressing, have improved the quality and consistency of glass products, enabling the production of glass-ceramics. The continuous innovation in glass has led to glass-ceramics, a class of materials bridging the properties of glasses and ceramics (Zhang and Liu, 2013). Recently, sustainable practices have emerged, with waste glass becoming a valuable precursor for glass-ceramics. This approach addresses environmental challenges while enabling the production of high-performance materials.

Glass-ceramics are fine-grained, uniform polycrystalline materials formed by controlled heat treatment of base glass (Dávalos *et al*., 2021; Deubener *et al*., 2018). They contain both glassy and crystalline phases, typically in ratios between 50 and 95% (Rahman, 2014; Aliyu, 2018). During heat treatment, one or more crystalline phases may precipitate, and their compositions differ from the parent glass (Aliyu, 2019). The main goal of producing glass-ceramics is to achieve superior properties, such as enhanced mechanical and chemical performance, which the original glass cannot provide (Ali *et al*., 2022).

Glass-ceramics can be prepared using conventional techniques, involving nucleation followed by crystal growth, or through sinter-crystallization, in which glass powders are compacted and heat-treated without a nucleating agent (Kang *et al*., 2017). Glasses of various compositions, including silicate, phosphate, and borate systems, have been used for glass-ceramics production (Rawlings *et al*, 2006). Aluminosilicate-based glasses are particularly suitable because they crystallize more readily under heat treatment. Crystallization transforms the glass structure from a randomly ordered network to a well-ordered, periodic crystalline structure (Ali *et al*., 2022). The microstructure of glass-ceramics typically contains 50 to 95% crystals dispersed in a residual glassy matrix (Aliyu, 2018). Controlling the crystallization process is essential to achieve uniform crystal distribution and desirable properties (Bahman and Mehdikhani, 2012).

The accumulation of waste window glass poses increasing environmental and health hazards, especially in regions with limited waste management infrastructure such as Nigeria. Recycling glass waste is therefore both environmentally necessary and economically beneficial. Alumina has been widely recognized for its positive effect on the chemical durability of glass (Zhang, 2011). This study aims to investigate the influence of gradual alumina (Al_2O_3) addition on the chemical and mechanical properties of glass-ceramics produced from waste window glass via the sinter-crystallization process.

2. MATERIALS AND METHOD

2.1 Experimental Procedures

Waste window glass pieces were collected locally, crushed, ground, and milled. The resulting powders were sieved using ASTM E11 standard mesh size of 100 microns. Representative samples were obtained from the bulk using the coning and quartering method. A portion of the glass powder was analysed by XRF to determine its oxide composition. Chemical-grade alumina (Al_2O_3) powders were then gradually added, and smooth compacts were formed with the aid of distilled water.

2.2 Formulation of Compacts

Six glass batches were prepared, as shown in Table 1, representing the base composition of six glass samples.

Table 1: Batch-Composition of Glass-Ceramic Samples

Batch	**Alumina (Al_2O_3) wt%**	**Glass powders mix wt%**
GC-1	0	100
GC-2	2	98
GC-3	4	96
GC-4	6	94

GC-5	8	92

Each batch was gradually modified by adding alumina (Al_2O_3) at the expense of waste glass powder. Alumina was added in varying amounts: 0%, 2%, 4%, 6%, 8%, and 10 wt%. Compacts were formed with the aid of distilled water. Each sample was placed in a 25 mm diameter mold and dry-pressed at 15 kN, producing green compacts with a diameter of 25 mm and thickness of 14 mm. The green compacts were air-dried at room temperature for 24 hours.

2.3 Heat-Treatment and Characterization of Samples

The sample compacts were heat-treated at 900°C in an electric furnace. The heating rate was 5°C per minute, and the samples were held for 1 hour to allow precipitation of crystalline phases within the residual glass matrix. After heat treatment, the samples were cooled gradually to room temperature. Phase analysis was performed using a PANalytical X'Pert Pro powder diffractometer with an X'celerator detector and Fe-filtered Co-Kα radiation. Each sample was scanned from 10° to 80° at a rate of 2° per minute.

2.3.1 Chemical durability test:

Ten samples were thoroughly dried, and the initial mass ($M_{initial}$) of each was recorded using an analytical balance. Solutions of 1 M sulfuric acid (H_2SO_4), 1 M nitric acid (HNO_3), and 1 M hydrochloric acid (HCl) were prepared. Each sample was immersed in the acid solutions at room temperature for 144 hours. After immersion, the samples were rinsed with distilled water to remove residual acid and left to dry at room temperature. Once dried, the samples were weighed again to determine their final mass (M_{final}). Acid resistance was calculated Equation 1:

$$\%\ weight\ loss = \frac{100 \times \left(M_{initial} - M_{final}\right)}{M_{initial}} \quad (1)$$

2.3.2 Compressive strength test

The compressive strength of the heat-treated samples was measured using a Universal Testing Machine (UTM). The dimensions of each sample were recorded with a meter rule. The UTM was calibrated, and samples were positioned centrally between the compression plates to ensure even load distribution and stability. A peak load was applied until fracture occurred. The peak load at fracture was recorded, and compressive strength was calculated using Equation 2.

$$\sigma c = PA\ \sigma c = PA \quad (2)$$

Where:
P = maximum load applied at failure (MN)
A = cross-sectional area of the sample in square meters m^2

2.3.3 Hardness test

The hardness of the heat-treated samples was measured using a Vickers Hardness Tester (MV1-PC). Each sample was placed on the stage, and a test load of 0.3 kgf was applied with a diamond pyramid indenter for 10 seconds. After indentation, an optical microscope was used to measure the diagonal and crack length. The test was repeated three times for each sample, and the average hardness was calculated.

3. RESULTS AND DISCUSSION

Table 2 shows the oxide composition of waste window glass powders determined by XRF analysis. The data indicate that the glass is a Na_2O-CaO-SiO_2 system. The composition consists of 59.801 wt% SiO_2, 23.300 wt% Na_2O, and 10.092 wt% CaO. No heavy metals were detected, suggesting that the heat-treated compacts are suitable for use in floor tiles (Garkida, 2007).

Table 2: Oxide composition of window glass powders by XRF

Oxides	Wt%
SiO_2	59.801
Na_2O	23.300

Al_2O_3	2.359
MgO	1.790
CaO	10.092
P_2O_5	0.064
SO_3	0.262
K_2O	0 38
Fe_2O_3	1.195
TiO_2	0.029
MnO	0.026

Figures 1 and 2 show the X-ray diffraction (XRD) analysis of samples GC-1 (0 wt% Al_2O_3) and GC-3 (4 wt% Al_2O_3). The differences in crystallinity between the two samples are mainly due to their alumina content. GC-1 precipitated 72.5 wt% wollastonite and 25.3 wt% quartz. In contrast, GC-3 exhibited higher crystallinity, with 80.5 wt% wollastonite and 17.5 wt% quartz. This indicates that adding alumina enhances overall crystallinity, promoting the formation of more crystalline phases such as wollastonite. The increased alumina in GC-3 likely improves mechanical and chemical properties, as well as structural stability and thermal resistance (Tulyaganov et al.,).

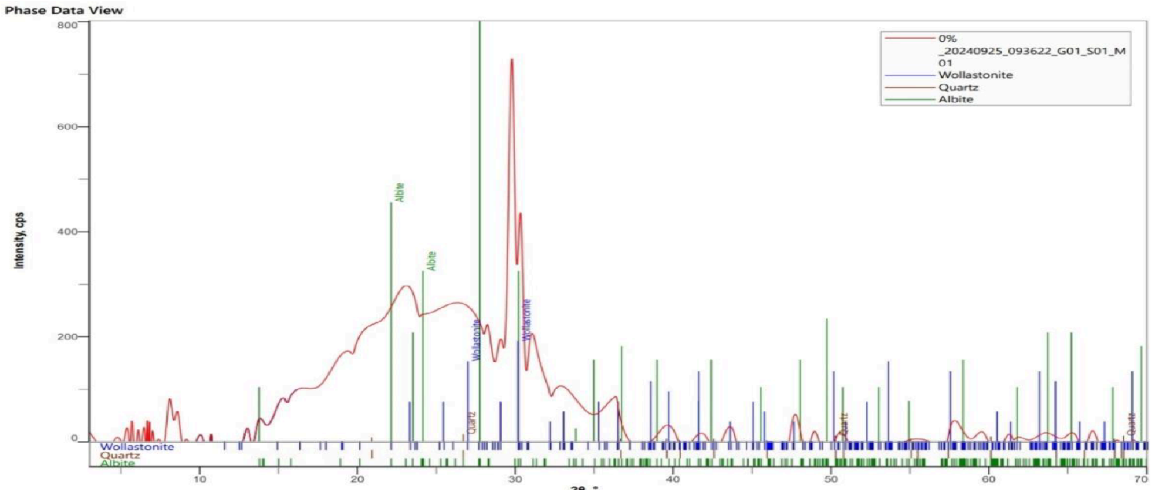

Figure 1: XRD analysis of sample GC-1 which has 0 wt% Al_2O_3

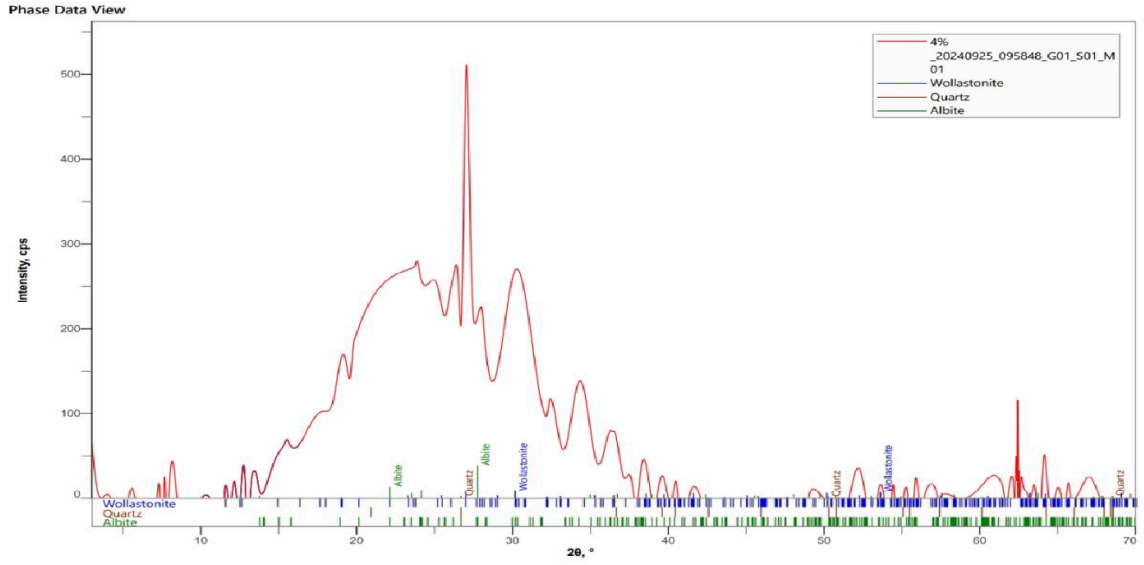

Figure 2: XRD analysis of sample GC-3 which has 4wt% Al_2O_3

GC-3 showed no shape distortion and appeared fully densified with a smooth surface, as shown in Plate 1. In contrast, GC-4 (6 wt% Al_2O_3) and GC-5 (8 wt% Al_2O_3) exhibited slight shape distortion, as seen in Plate 2.

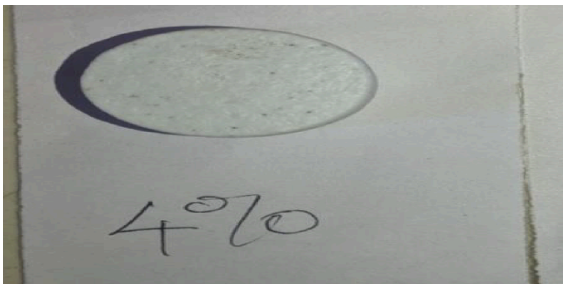

Plate 1: Sample GC-3 (4 wt% Al_2O_3) showed no shape distortion after heat treatment at 900°C.

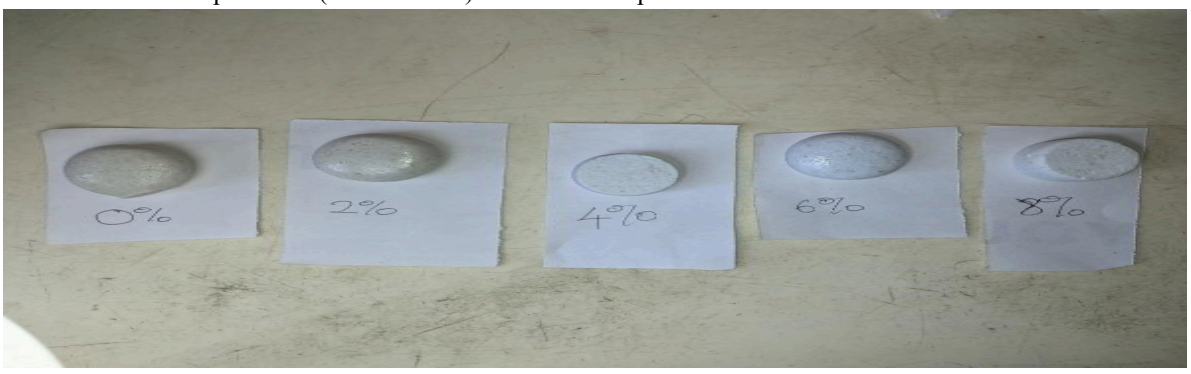

Plate 2: Samples GC-1, GC-2, GC-3, GC-4, and GC-5 after sintering at 900°C.

Acids generally attack the fluxing agents (Na_2O, K_2O) in glass by substituting H^+ ions for the alkali ions, which opens the silica network (Salah, 2016). The chemical solubility of glass-ceramic samples (GC-1 to GC-5, 0–8 wt% Al_2O_3) was evaluated in 1 M solutions of HCl, H_2SO_4, and HNO_3. Each sample was immersed for 24 hours, and weight loss was measured before and after immersion. The results are presented in Figure 3.

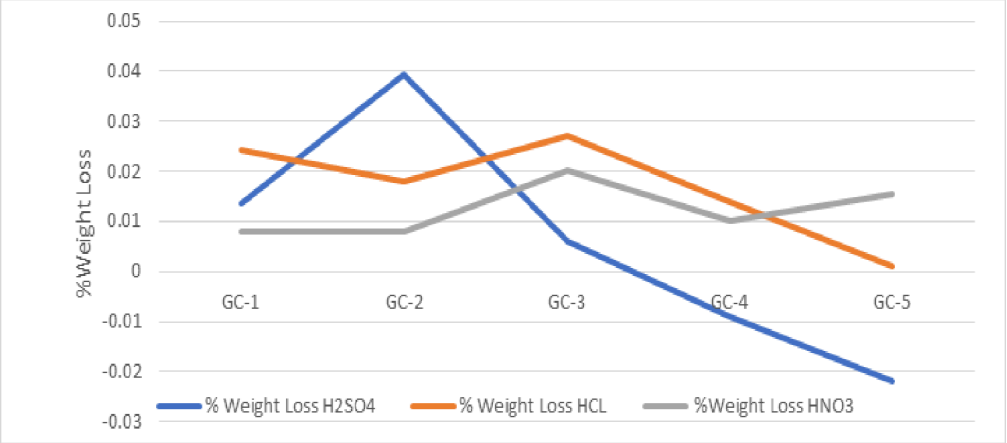

Figure 3: Effect of gradual Al_2O_3 addition on the chemical durability of glass-ceramic samples in 1 M HCl, 1 M H_2SO_4, and 1 M HNO_3 solutions.

In H_2SO_4, weight loss peaked for GC-2 (2 wt% Al_2O_3) but decreased sharply with higher alumina content, showing strong resistance in GC-4 (6 wt% Al_2O_3) and GC-5 (8 wt% Al_2O_3). In HCl, weight loss remained relatively stable, with GC-5 performing best. HNO_3 caused minimal weight loss across all samples, with GC-1 slightly more affected. These results indicate that alumina significantly enhances chemical stability and resistance to corrosive environments (Demirkesen and Göller, 2003). Overall, GC-5 (8 wt% Al_2O_3) exhibited the highest resistance, particularly to H_2SO_4, likely due to increased crystallinity and the formation of wollastonite, a phase known for acid resistance (Sun *et al*., 2011). GC-1, without alumina, was the least resistant.

The compressive strength results of the glass-ceramic samples are shown in Figure 4. GC-1, GC-2, GC-3, and GC-5 demonstrated a clear correlation between alumina content and compressive strength. GC-1, without alumina, had the lowest strength at 11.61 MPa, indicating low resistance to compressive forces. GC-2 (2 wt% Al_2O_3) also showed low strength at 10.59 MPa. In contrast, GC-3 (4 wt% Al_2O_3) and GC-5 (8 wt% Al_2O_3) exhibited significantly higher strengths of 18.57 MPa and 22.59 MPa, respectively. This trend aligns with previous studies showing that alumina enhances microstructural integrity, reduces microcracks, and increases overall strength (Sun *et al*., 2014).

Sample GC-4 was not tested due to irregular dimensions, making it unsuitable for uniaxial compression. The higher alumina content in GC-3 and GC-5 makes them suitable for applications requiring high load-bearing capacity. GC-5 showed the highest compressive strength, confirming its suitability for demanding applications (Aliyu and Salisu, 2023). When tested under an elevated load of 25 kN, GC-5's strength slightly decreased to 19.74 MPa, likely due to stress-induced microstructural changes. Variations in strength across samples may result from differences in density, porosity, and alumina distribution. GC-1 is better suited for low-strength applications, while GC-2, GC-3, and GC-5 are suitable for higher structural demands (Binhussain *et al*., 2014).

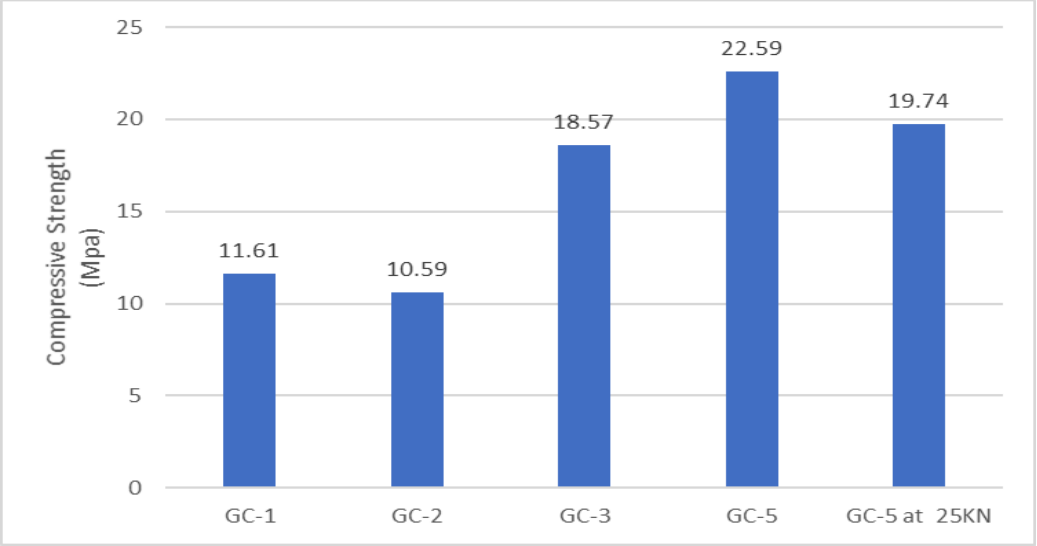

Figure 4: Effect of gradual replacement of waste glass powders by Al_2O_3 on the compressive strength of glass-ceramics samples

The hardness of glass-ceramics depends on the bond strength of its composition and the packing density of ions in the structure (Aliyu, 2018). Figure 5 shows the effect of alumina (Al_2O_3) addition on the hardness of the samples. GC-1 (0 wt% Al_2O_3) exhibited low hardness after heat treatment, indicating weaker resistance.

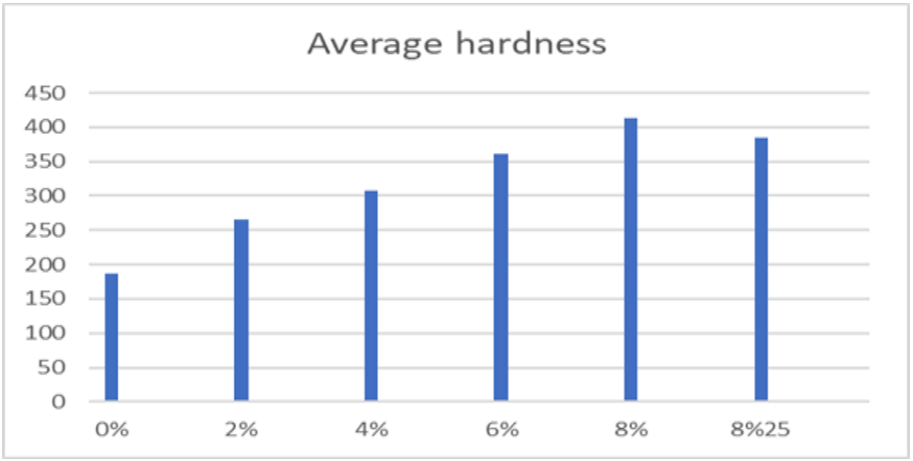

Figure 5: Effect of alumina addition to hardness on various glass-ceramics samples.

Hardness increased noticeably from GC-2 to GC-5 as the alumina content gradually increased. This trend is expected because converting glass into glass-ceramics typically increases hardness. The improvement is attributed to the formation of more wollastonite in the samples from GC-2 to GC-5.

4. CONCLUSION

This study highlights the potential of developing novel glass materials from waste glass. It demonstrates that reactive sinter crystallization can produce glass-ceramics from soda-lime-silica–based glass. Window glass, as an example, can be crystallized without a nucleating agent by using alumina as a crystallization promoter. The precipitated crystalline phases—wollastonite, quartz, and albite—are dispersed in the residual glass matrix and increase with gradual alumina addition. In GC-3, the addition of 4 wt% alumina resulted in 80.5 wt% wollastonite and 17.5 wt% quartz in the matrix. The higher alumina content enhances crystallization, improving structural stability, mechanical strength, and chemical resistance. These results indicate that alumina addition significantly improves overall crystallinity and the performance of glass-ceramic materials.

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