Sinter Crystallization of Soda Lime Silicate Waste Glass Modified With EggshellDerived CaO for Glass Ceramic Tile Applications

Abstract

The utilization of post-consumer and industrial wastes for the production of glass ceramics provides a sustainable pathway for developing high performance construction materials. In this study, glass-ceramic tiles were synthesized via the sinter crystallization technique using waste soda lime silicate (TV panel) glass and eggshell-derived calcium oxide (CaO) as alternative raw materials. The waste glass served as a silica rich matrix, while eggshell waste acted as a low cost and sustainable CaO source. Raw materials and sintered products were characterized using X-ray fluorescence (XRF), X-ray diffraction (XRD), Fourier transform infrared spectroscopy (FTIR), and scanning electron microscopy coupled with energy dispersive spectroscopy (SEM/EDS). Compacted samples were sintered at 650, 700, and 750 °C, and their phase composition, microstructure, density, mechanical properties, abrasion resistance, and chemical durability were evaluated.XRD analysis revealed partial crystallization within a residual glassy matrix, with quartz and calcite identified as the dominant crystalline phases. Increasing the sintering temperature promoted crystallization and densification up to 700 °C, as confirmed by SEM observations. The sample sintered at 700 °C exhibited optimal performance, achieving the highest compressive strength (28.54 MPa), enhanced density, improved hardness, superior abrasion resistance, and excellent chemical durability in acidic and alkaline environments. These results demonstrate that waste glass and eggshell derived CaO can be effectively utilized to produce durable glassceramic tiles suitable for sustainable building applications.

***Keywords**: Characterization, Eggshellderived CaO, Glass ceramics, Mechanical properties, Sinter crystallization, Waste glass.*

1. Introduction

The growing demand for sustainable construction materials has intensified research into the valorization of industrial and post-consumer wastes as alternative raw materials for advanced ceramics.Although nature has provided essential components for the manufacture of glasses with abundant and easily removable components for centuries, synthetic chemical products and a wide range of solid waste are also used today (Rawlings *et al*., 2006). Waste recycling has become an unavoidable requirement to optimize the use of natural resources (Rincon, 2016). Furthermore, by using wastes as alternative raw materials, it is possible to obtain new materials with added value and mitigate the environmental risk (Rincon, 2016; Barbieri *et al*., 2000; Saparuddin *et al*., 2020).However, the use of waste as raw material requires strict control over the chemical composition, since the mixtures must contain the typical components of stable glasses, in particular Si and Al oxides and nucleating agents that favor crystallization. In this way, each residue must contribute an appropriate amount of vitrifying agents such as SiO_2 and Al_2O_3, as well as elements that modify and promote fusion ($Na_2O_3K_2O$), in addition to stabilizing agents such as CaO and MgO, which are the components that give rise to glasses with adequate or special characteristics (Barbieri *et al.*, 2000; Karamanov *et al.*, 2003).Among these wastes, soda lime silicate glass constitutes a major fraction of municipal solid waste streams due to its extensive use in packaging, glazing, and electronic applications. Although glass is theoretically recyclable, large quantities of waste glass remain underutilized because of contamination, color mixing, and economic constraints, leading to significant environmental burdens (Yang *et al*., 2014). Converting waste glass into value added glass ceramic products represents an effective strategy to mitigate disposal challenges while producing materials with enhanced functional properties.

Glass ceramics are polycrystalline materials produced through the controlled crystallization of parent glasses and are characterized by a combination of crystalline phases embedded within a residual glassy matrix(Deubener *et al.*, 2018). This unique microstructure enables glass ceramics to exhibit superior mechanical strength, chemical durability, abrasion resistance, and thermal stability compared with conventional glasses (Holand & Beall, 2019).Consequently, glass ceramics have found widespread applicationsdue to their characteristics, such as chemical and mechanical durability, surface hardness, low thermal expansion coefficient, which are easily adapted to practical usesin architectural tiles, flooring materials, countertops, and other load bearing construction components (Mauro & Zanotto, 2014). Among the various processing routes, the sinter crystallization technique has attracted considerable attention for waste derived glassceramics, as it allows densification and crystallization to occur simultaneously from compacted glass powders at relatively lower temperatures, reducing energy consumption and processing costs(Karamanov & Pelino, 2006; Bernardo & Colombo, 2006).

The incorporation of calcium rich additives plays a critical role in tailoring the crystallization behavior and properties of soda lime silicate glass systems. Calcium oxide (CaO) acts as a network modifier, influencing viscosity, nucleation kinetics, and phase development during heat treatment(Shelby, 2005; Zanotto & Mauro, 2017). Conventionally, CaO is introduced using high-purity chemical reagents or natural limestone; however, these sources are associated with additional cost and environmental impacts. In this context, waste eggshells have emerged as an attractive, low-cost, and sustainable alternative CaO source. Eggshells are primarily composed of calcium carbonate ($CaCO_3$), which decomposes to CaO upon heating, and are generated in large quantities by households, restaurants, and food-processing industries. Their reuse not only diverts biodegradable waste from landfills but also aligns with circular economy principles(Onwubu *et al.*, 2019; Arshad *et al.*, 2021).

Recent studies have demonstrated the feasibility of utilizing eggshell derived CaO in cementitious materials, bio ceramics, and glass formulations, where it contributes to improved densification, phase formation, and mechanical performance(Onwubu *et al*., 2019; Arshad *et al*., 2021). However, investigations focusing on the modification of waste soda lime silicate glass with eggshell derived CaO for glass ceramic tile applications remain limited. In particular, there is a need to better understand how CaO addition influences sinter crystallization behavior, phase evolution, microstructure, and key performance indicators such as mechanical strength and chemical resistance at relatively low sintering temperatures.

Therefore, the present study investigates the sinter crystallization of soda lime silicate waste glass modified with eggshell derived CaO for the development of glass ceramic tiles. Emphasis is placed on the role of eggshell derived CaO in promoting crystallization, microstructural refinement, and durability of the resulting glass ceramics. By combining two abundant waste streams, waste glass and eggshells this work aims to demonstrate a sustainable and cost effective route for producing high performance glass ceramic materials suitable for tiling and other building applications

2.0 Materials and Methods

2.1 Raw Materials

Waste eggshells and soda lime silicate television panel glass were employed as the primary raw materials. The waste TV panel glass was sourced from discarded and damaged television screens collected from local television repair workshops within Funtua Local Government Area, Katsina

State, Nigeria. This type of glass represents a significant component of electronic waste (e-waste) streams, as it is commonly generated during screen replacement and repair activities and is often disposed of without formal recycling pathways. The eggshells were obtained from chicken restaurants in Funtua, where large quantities are generated daily as food processing waste. Polyvinyl acetate (PVAc) was employed as a temporary binder. Prior to processing, all raw materials were thoroughly washed with distilled water to remove surface contaminants.

2.2 Sample Preparation

The eggshells were cleaned, air dried for 24 h, and oven dried at 110 °C for 3 h to eliminate residual moisture. The dried eggshells were crushed and ground into powder. Waste soda lime silicate glass were also crushed using a jaw crusher and subsequently milled in a planetary ball mill for 24 h at 50 rpm to obtain a homogeneous fine powder. Both powders were sieved to a particle size of 75 µm, consistent with prior studies indicating enhanced sintering and densification behavior at reduced particle sizes.

2.3 Characterization of Raw Materials

Chemical composition of the eggshell and soda lime glass powders ware determined using Energy Dispersive X-ray Fluorescence (EDXRF) (Thermo Scientific QUANT'X 9952120), following ASTM D8064-16. Phase identification and structural analysis were carried out by X-ray Diffraction (XRD) using a Rigaku Miniflex diffractometer with Cu Kα radiation ($\lambda = 1.5418$ Å), operated at 30 kV and 15 mA, over a 2θ range of 5-70°, in accordance with ASTM E3294-22.Microstructural features and elemental distribution were examined using Scanning Electron Microscopy coupled with Energy Dispersive Spectroscopy (SEM/EDS) (Phenom ProX), following the ASTM E1508-12a. Functional groups and bonding characteristics were investigated using Fourier Transform Infrared Spectroscopy (FTIR), in accordance with ASTM E1252-98.

3.0.Results and Discussion

3.1 Results

Table 1present the XRF results of the eggshell powder (ES) and waste glass (WG). The eggshell powder is dominated by CaO (85.84 wt. %), inorganic impurities and traces ware also observed in the Eggshell powder.This high lime CaO content agrees with previous studies reporting eggshells as calcium rich materials suitable for ceramic applications (Arshad *et al*., 2021; Shiferaw *et al*., 2019).The waste soda lime silicate glass is characterized by a high SiO_2 content (61.04 wt. %), together with modifiers and intermediate oxides. These results are consistent with earlier reports on waste container and TV screen glasses (Diaz *et al*., 2015; Kazmi *et al*., 2021). The combined CaO rich eggshell and silica rich waste glass compositions are favorable for glass ceramic formation via sinter crystallization.

Table1. Shows the Results of waste Soda Lime Silica Glass Using of X-ray Fluorescence

Elements (Wt. %)	SiO_2	Na_2O	CaO	K_2O	Al_2O_3	MgO	Fe_2O_3	PbO	BaO	SrO	Trace
Waste glass	61.04	13.25	2.14	2.81	2.53	2.96	0.17	2.50	1.90	ND	9.17
Eggshell	1.24	3.16	85.84	0.26	2.39	2.58	0.17	ND	0.46	3.46	0.43

3.2. Microstructural analysis

XRD analysis of the eggshell powder revealed calcite ($CaCO_3$) as the dominant crystalline phase, with characteristic peaks at 2θ, 29°, 47°, and 48°, alongside minor phases of chlorite, albite, and quartz as presented in figure 1. Quantitative phase estimation indicated approximately 80% calcite, confirming eggshell as a calcium rich precursor, consistent with literature reports. In contrast, the waste soda lime glass exhibited a broad diffuse halo without sharp diffraction peaks, confirming its amorphous nature, which is typical of glassy materials as depicted in figure 2.FTIR spectra of the eggshell powder presented in figure 3. Showed prominent carbonate-related bands at 1401, 715, and 872 cm^{-1}, corresponding to $CaCO_3$, as well as O-H vibrations associated with adsorbed moisture. The waste glass FTIR spectrum was dominated by silicate network vibrations, with bands at 760 cm^{-1} (Si-O) and 969 cm^{-1} (Si-O-Si asymmetric stretching), confirming its silicate based composition. These observations agree well with previous studiesby Onwubu *et al*., (2019) who conducted their study on In vitro evaluation of nanohydroxyapatite synthesized from eggshell waste in occluding dentin tubules.SEM micrographs of the eggshell powder revealed irregular, porous particles with non-uniform size distribution, while EDS confirmed calcium as the major element, with minor Na, Al, Mg, and trace elements. The waste glass powder displayed angular and fragmented particles typical of mechanically crushed amorphous glass; EDS analysis showed Si as the dominant element, with Na, Ca, Al, Mg, and K as secondary constituents.

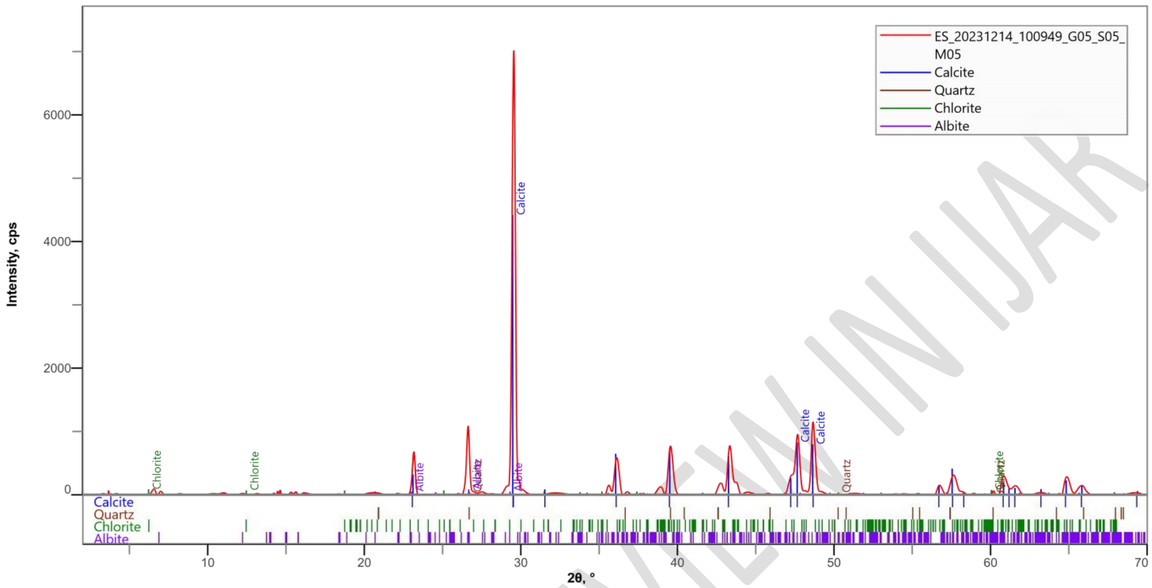


Figure 1: X-ray Diffraction of Eggshell Powder

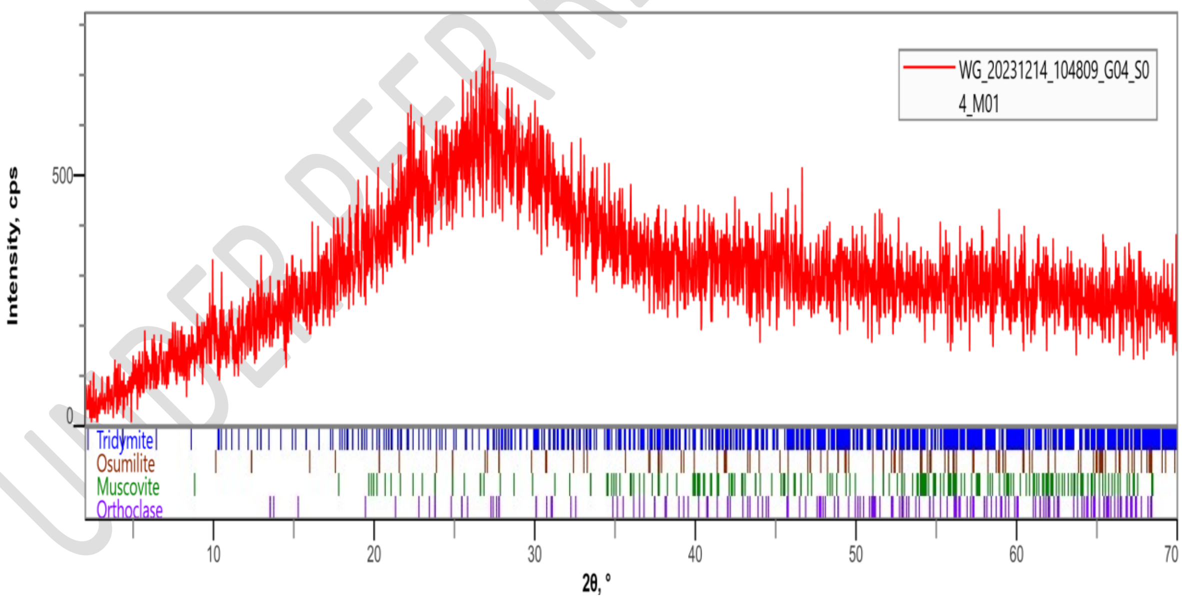


Figure 2: X-ray Diffraction of Glass Powder

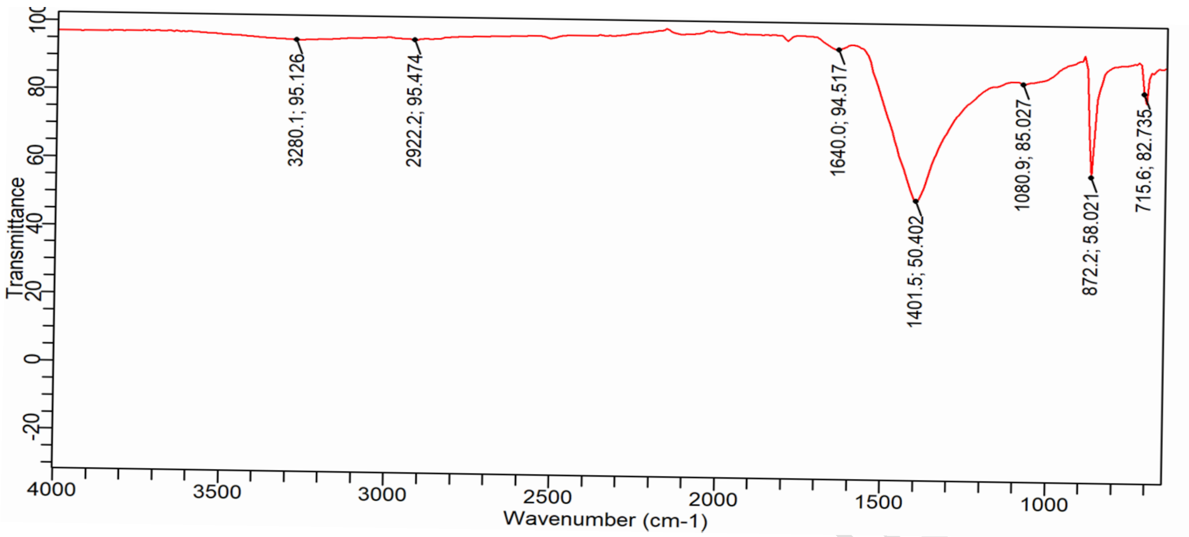


Figure 3: FT-IR of Eggshell Powder

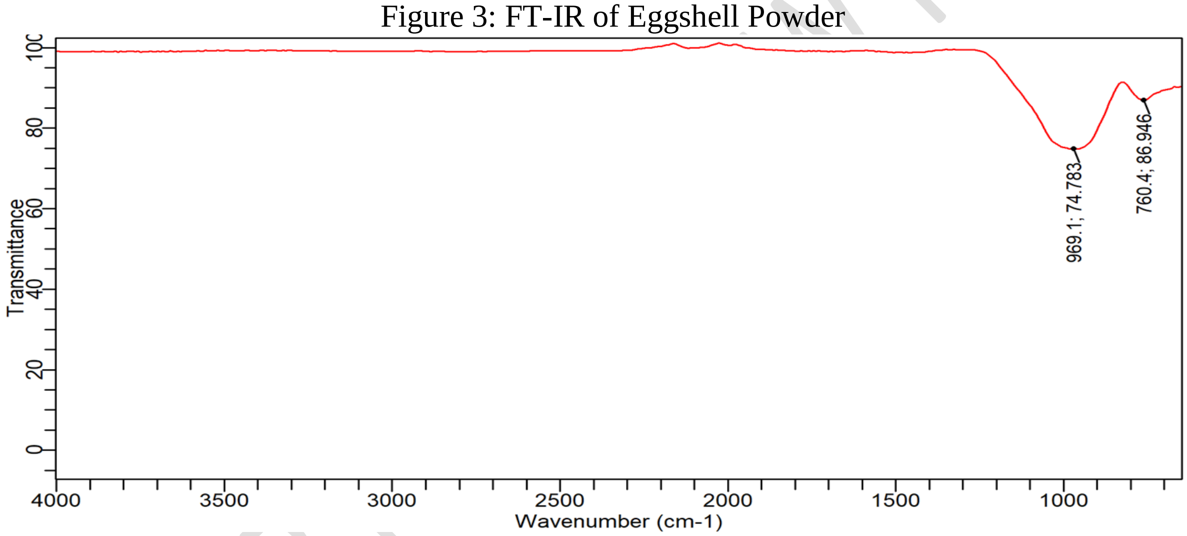
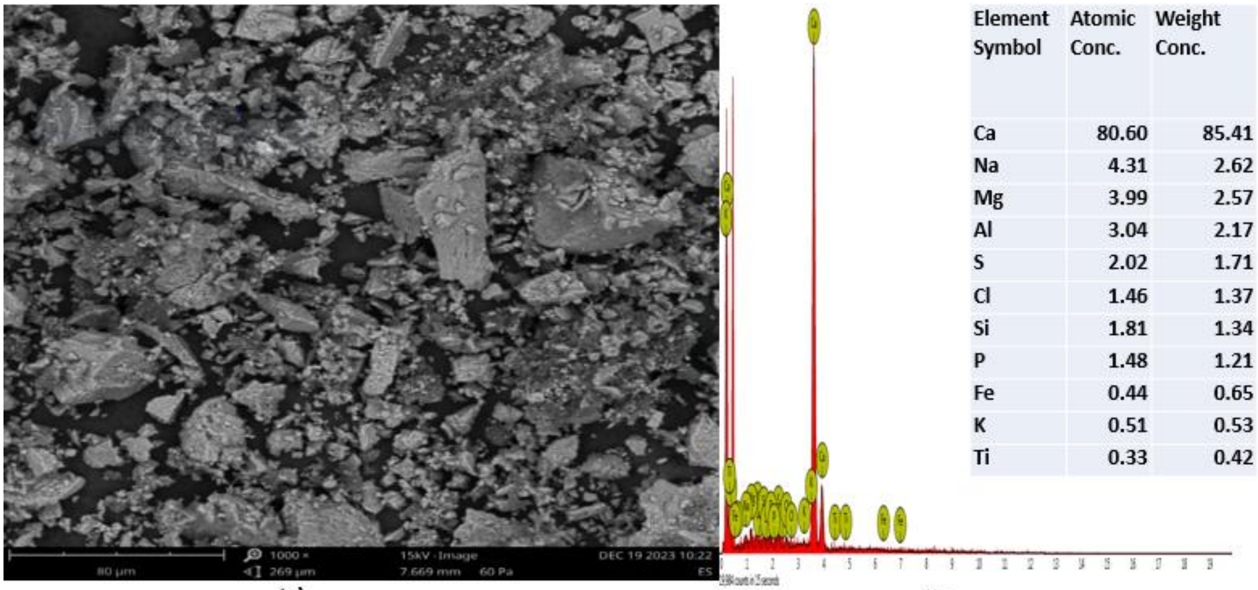


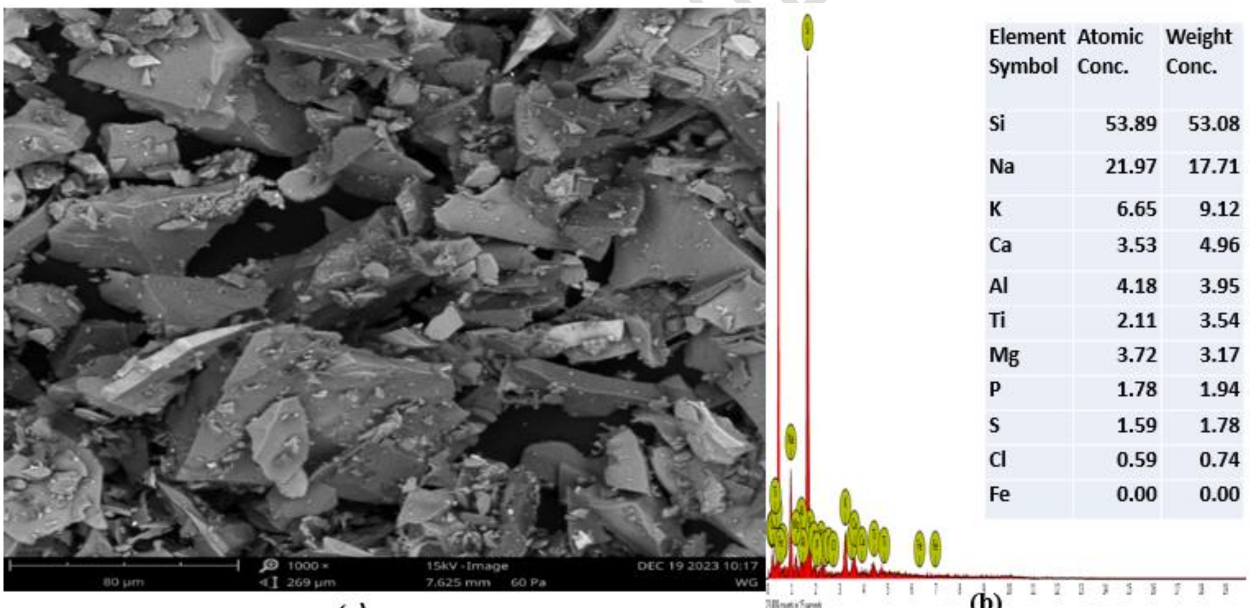
Figure 4: FT-IR of Waste Glass Powder



Element Symbol	Atomic Conc.	Weight Conc.
Ca	80.60	85.41
Na	4.31	2.62
Mg	3.99	2.57
Al	3.04	2.17
S	2.02	1.71
Cl	1.46	1.37
Si	1.81	1.34
P	1.48	1.21
Fe	0.44	0.65
K	0.51	0.53
Ti	0.33	0.42

(a) (b)

Figure 5: (a) SEM (b) EDS micrographs of Eggshell powder



Element Symbol	Atomic Conc.	Weight Conc.
Si	53.89	53.08
Na	21.97	17.71
K	6.65	9.12
Ca	3.53	4.96
Al	4.18	3.95
Ti	2.11	3.54
Mg	3.72	3.17
P	1.78	1.94
S	1.59	1.78
Cl	0.59	0.74
Fe	0.00	0.00

(a) (b)

Figure 6: (a) SEM (b) EDS micrographs of Waste Glass

3.3. Characterization of Glass Ceramics Samples

3.3.1 X-ray Diffraction (XRD) Analysis

The crystalline phases developed in the glass ceramic samples during controlled sinter crystallization were identified by X-ray diffraction (XRD) using a Rigaku Miniflex diffractometer with Cu Kα radiation over a 2θ range of 5 -70°. Phase identification was performed using ICDD PDF-4 database files. The XRD patterns revealed that the sintered glass ceramics consisted of a predominant amorphous matrix with superimposed crystalline peaks, indicating partial crystallization.The diffraction patterns showed the presence of quartz (SiO_2) as the main crystalline phase, while wollastonite ($Ca_3Si_3O_9$), which is commonly reported in CaO SiO_2 systems, was not detected. The absence of wollastonite is attributed to the high SiO_2 content of the waste glass, the relatively low CaO contribution from eggshell derived additives, and the comparatively low sintering temperatures employed. Similar observations have been reported in previous studies, where wollastonite formation required higher CaO contents and sintering temperatures close to 900 °C (Soares *et al*., 2018).At 650 °C, only weak diffraction peaks corresponding to $CaCO_3$ were observed, indicating limited crystallization within an amorphous glassy phase. Increasing the sintering temperature to 700 °C resulted in the appearance and growth of calcite ($CaCO_3$) as the dominant crystalline phase. Further heating to 750 °C led to enhanced crystallization, characterized by increased intensities of calcite and quartz peaks and a reduction in the amorphous background. These temperature-dependent crystallization trends are consistent with reports by (Salman *et al*., 2017; Mugani*et al*.,2015) for similar glass-based systems.

Semi quantitative phase analysis revealed that the relative contents of quartz, calcite, muscovite, and orthoclase varied systematically with sintering temperature, with quartz content increasing at higher temperatures. The overall degree of crystallinity was evaluated using peak area analysis based on the ratio of crystalline to total (crystalline + amorphous) contributions, confirming that increasing sintering temperature promotes crystallization while retaining a residual glassy phase. This controlled phase evolution is essential for tailoring the microstructure and performance of glass ceramics intended for tile applications.

3.3.2 Scanning Electron Microscopy and Energy Dispersive X-ray spectroscopy (SEM/EDS) the morphology and size of crystals in the glass ceramics were identified by the phenom pro X 800-07334 scanning electron microscopeCompanyLimited, from Netherland.The sinter crystallized glass ceramics demonstrated temperature dependent microstructural evolution. Samples sintered at 650 °C exhibited heterogeneous morphology with open porosity and partial particle bonding. Increasing the sintering temperature from 650 to 750 °C led to enhanced particle coalescence, reduced pore size distribution, and improved densification. This microstructural refinement is directly associated with improved mechanical performance, including increased compressive strength, hardness, and abrasion resistance, confirming the suitability of the developed glass ceramics for tiling applications.

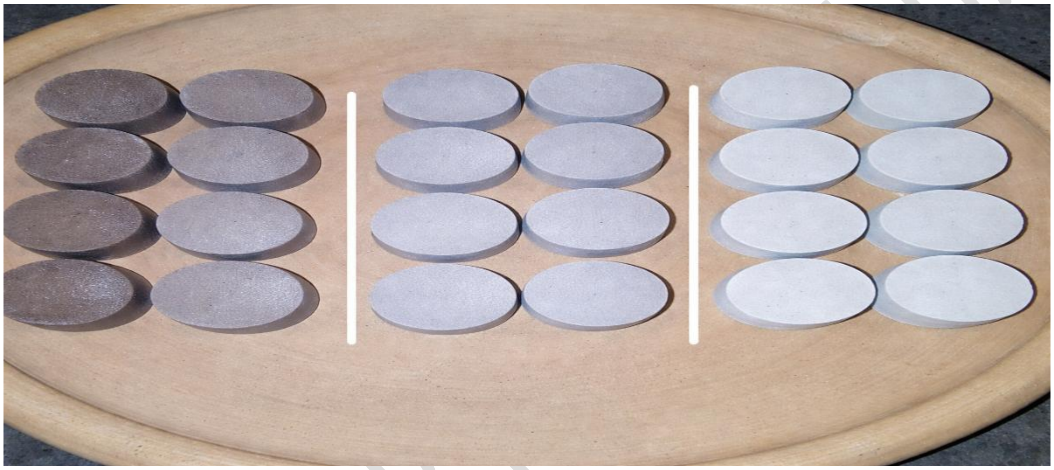


Plate 1: Sinter Crystallized Glass Ceramics

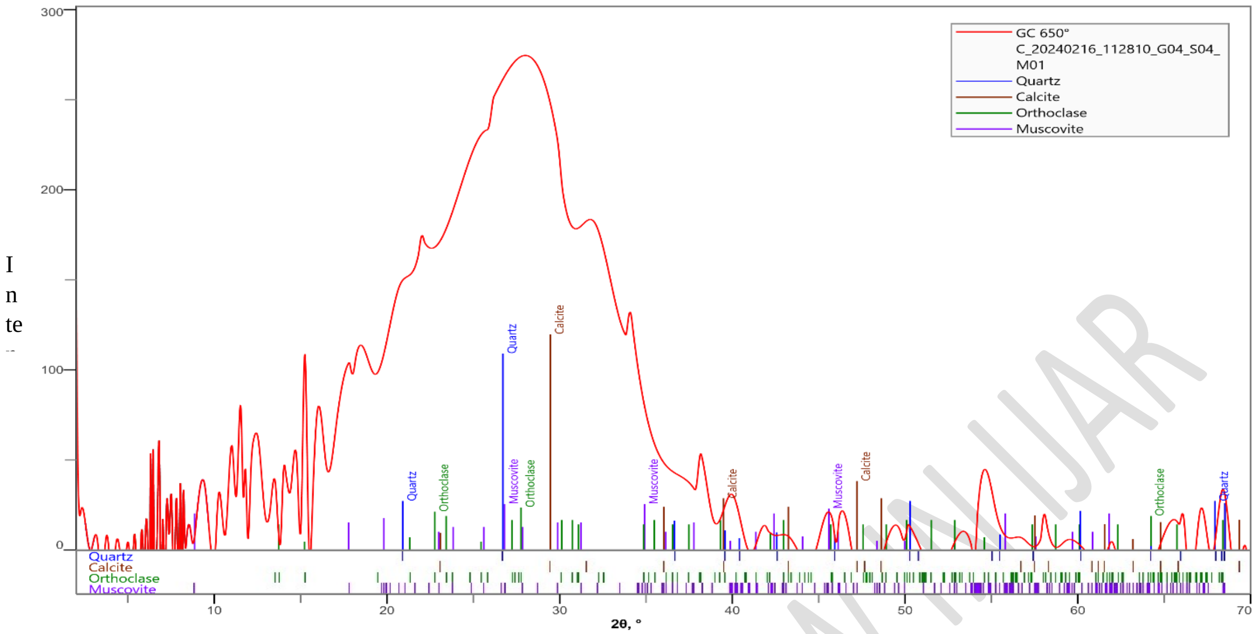


Figure 7: XRD result of Glass Ceramics Sintered at 650 ºC

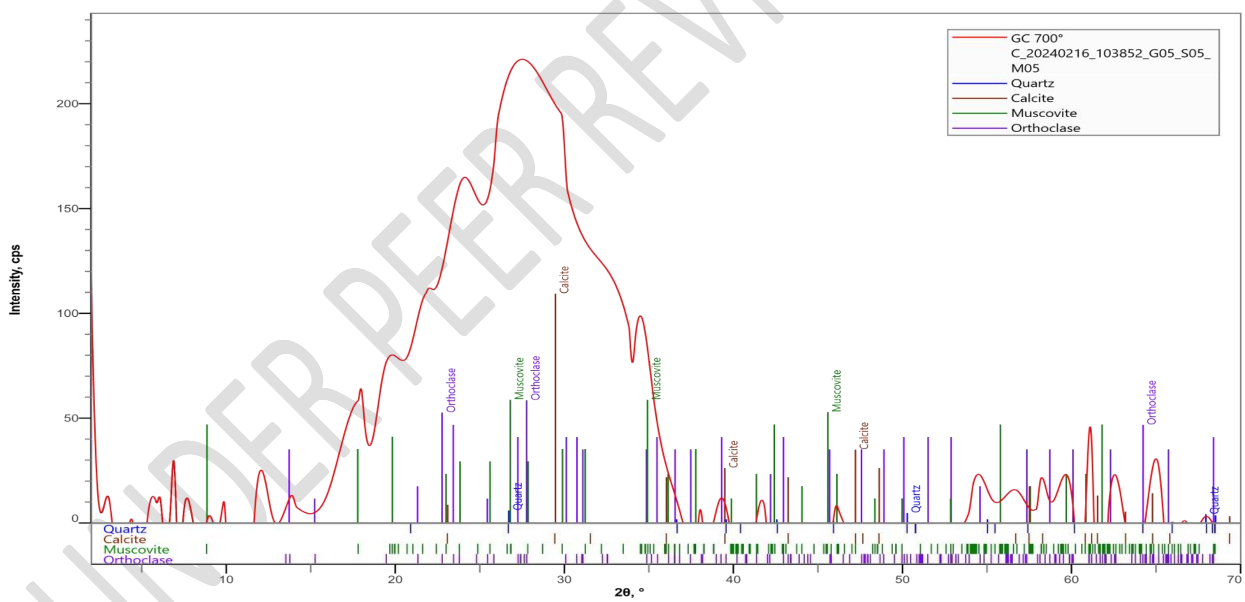


Figure 8: XRD result of Glass Ceramics Sintered at 700 ºC

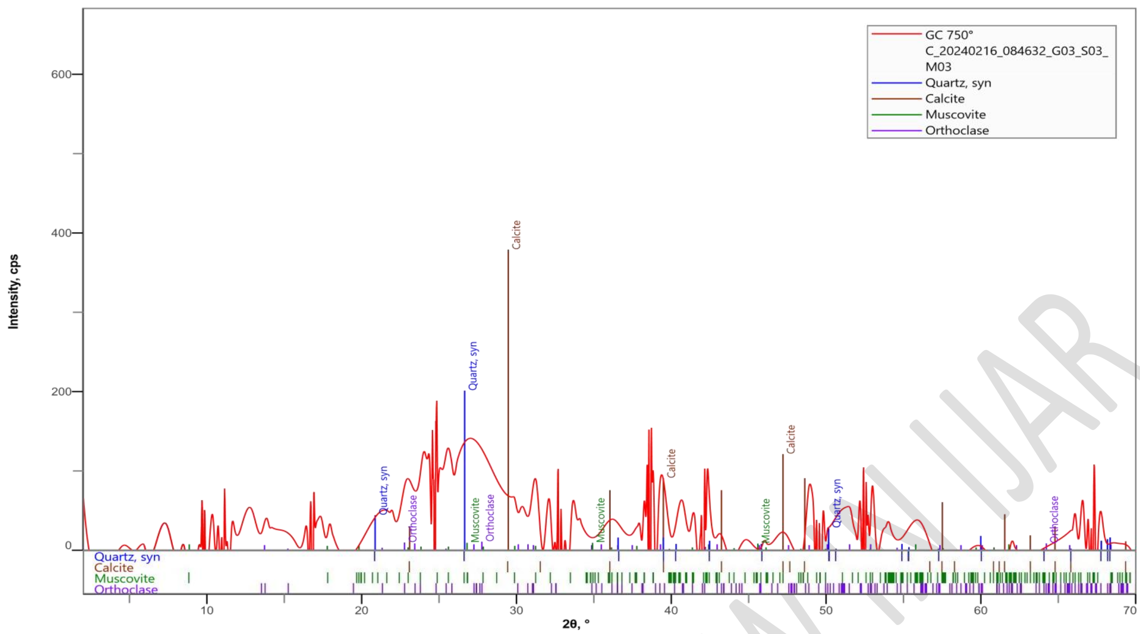
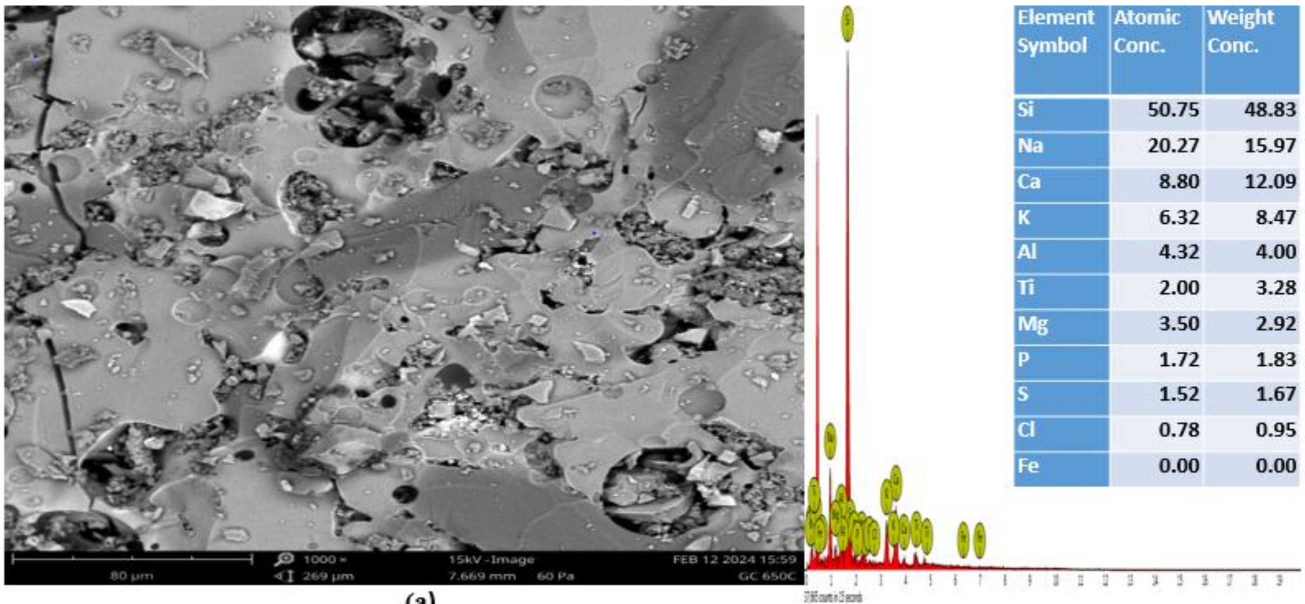


Figure 9: XRD result of Glass Ceramics Sintered at 750 ºC

Table 2.Crystallinity index

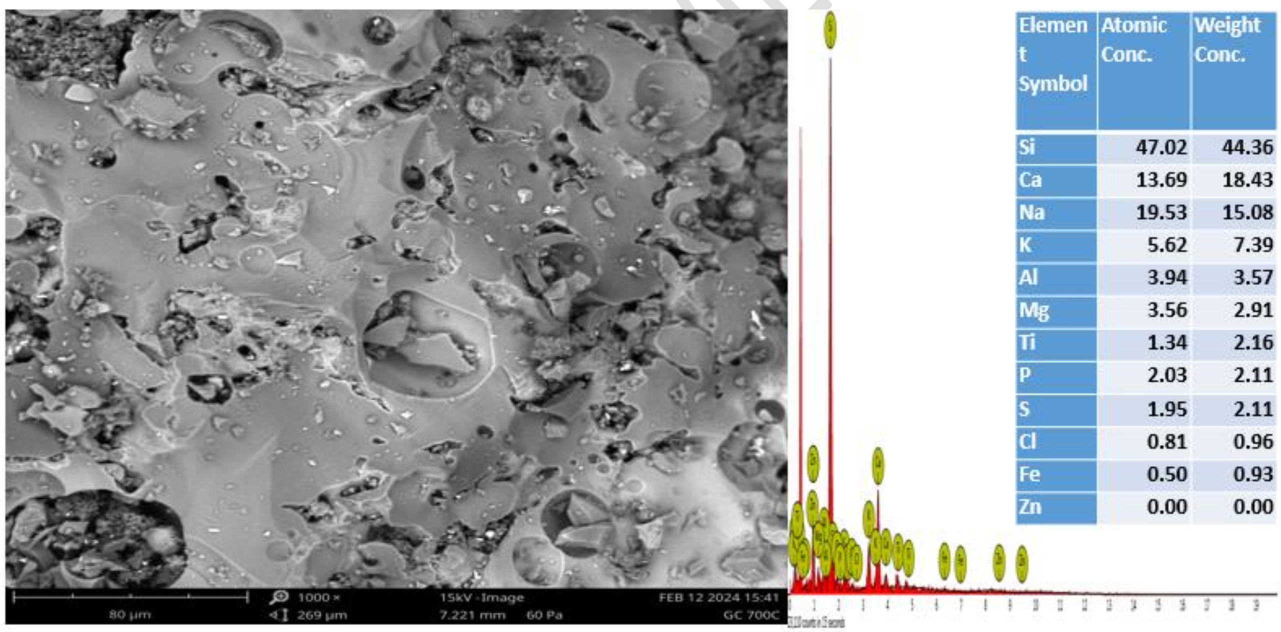
Temperatures		**Mineral phases**		
ºC	Quartz (%)	Calcite (%)	Muscovite (%)	Orthoclase (%)
650 ºC	31.7	19	30	19.3
700 ºC	35.2	10.7	20.4	33.8
750 ºC	44	15	14	27



Element Symbol	Atomic Conc.	Weight Conc.
Si	50.75	48.83
Na	20.27	15.97
Ca	8.80	12.09
K	6.32	8.47
Al	4.32	4.00
Ti	2.00	3.28
Mg	3.50	2.92
P	1.72	1.83
S	1.52	1.67
Cl	0.78	0.95
Fe	0.00	0.00

(a) (b)

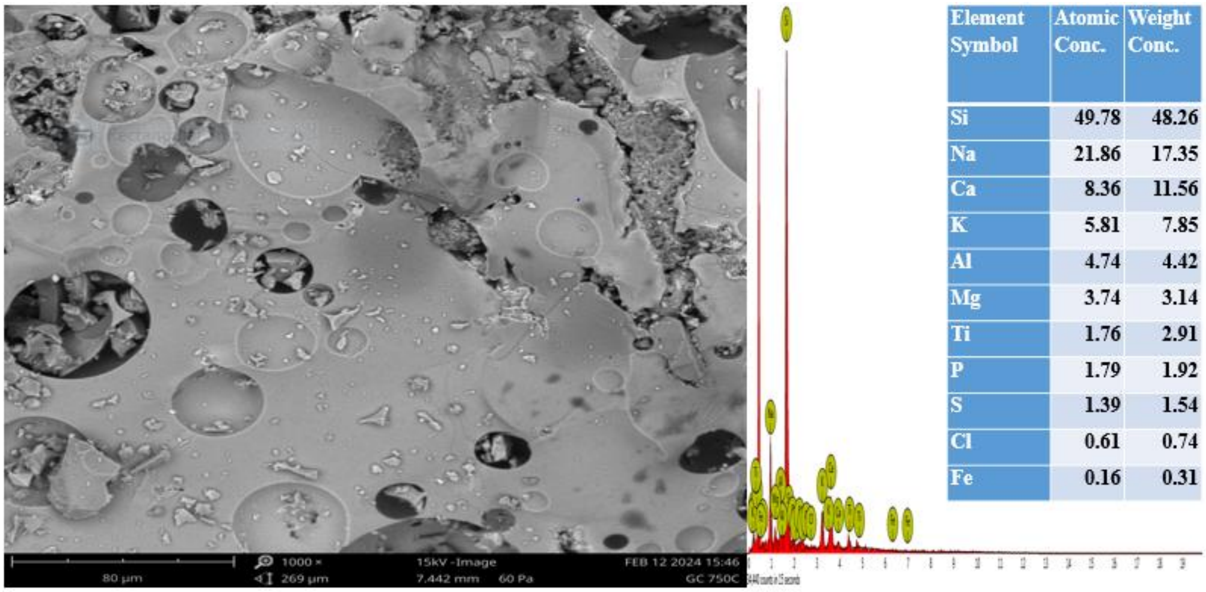
Figure 10: (a) SEM (b) EDS micrographs of Glass Ceramics Sintered at 650ºC



Element Symbol	Atomic Conc.	Weight Conc.
Si	47.02	44.36
Ca	13.69	18.43
Na	19.53	15.08
K	5.62	7.39
Al	3.94	3.57
Mg	3.56	2.91
Ti	1.34	2.16
P	2.03	2.11
S	1.95	2.11
Cl	0.81	0.96
Fe	0.50	0.93
Zn	0.00	0.00

(a) (b)

Figure11: (a) SEM (b) EDS micrographs of Glass Ceramics Sintered at 700ºC



Element Symbol	Atomic Conc.	Weight Conc.
Si	49.78	48.26
Na	21.86	17.35
Ca	8.36	11.56
K	5.81	7.85
Al	4.74	4.42
Mg	3.74	3.14
Ti	1.76	2.91
P	1.79	1.92
S	1.39	1.54
Cl	0.61	0.74
Fe	0.16	0.31

(a) (b)

Figure 12: (a) SEM (b) EDS micrographs of Glass Ceramics Sintered at 750ºC

3.4. Compressive Strength Test Result

The compressive strength of the glass ceramicssintered at 650ºC shown in plate Iwas 27.44 MN/m^{-2}which increased when the sample sintered at 700ºC reaching a value of 28.54 MN/m^{-2}, clearly showing an increase in mechanical strength and also decreases when the temperature increases to 750 ºC reaching a value of 13.34 MN/m^{-2}this is attribute to the distortion of shape and expansion at 750 ºC, and result in decreases of mechanical strength. One of the factors that influence this aspect is the porosity, in which the mechanical properties decrease with the increase of the porosity according to the generalized mixing rule, aspect correlated and demonstrated according to the data presented in Figure13, this feature is also associated with a 3.5% increase in the Quartz phase and a 14.5% increase in the orthoclase phase, and 8.3% decrease in the Calcite phase and a 9.6% decrease in the Muscovite phase. In addition to the greater cohesion between particles according to the morphology presented in Figure 11, in general it could be established that the based glass-ceramic materials, depending on the crystallization temperature, present increased mechanical resistance. Therefore, the higher the density, the lower the porosity, and the greater the Compressive strength of the glass ceramics materials.

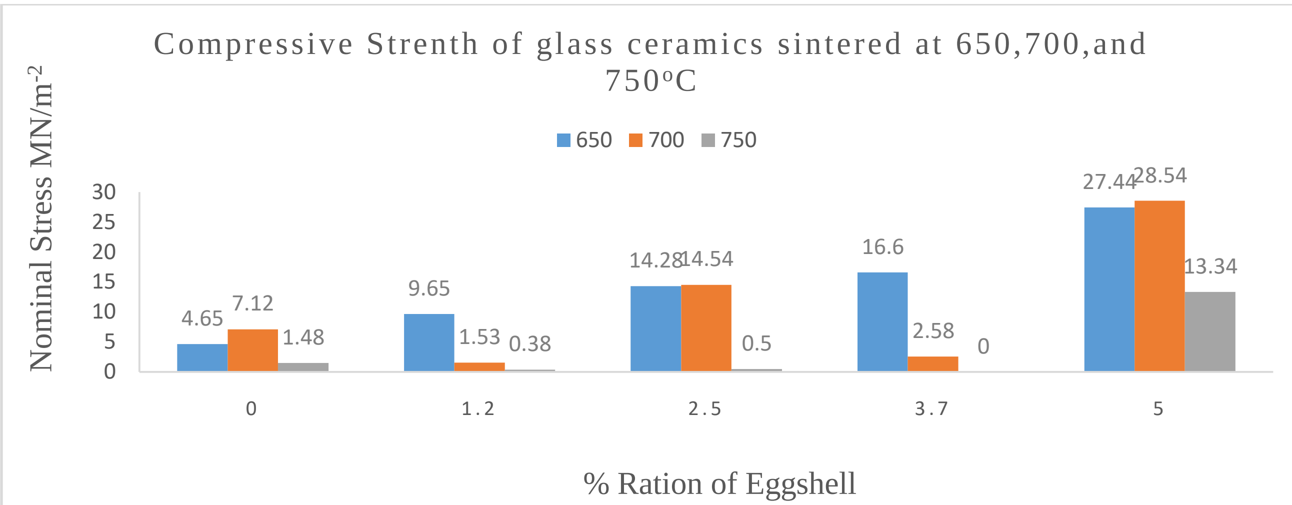


Figure 13: Compressive Strength of the sintered glass ceramics

3.5. Vickers Hardness Test Result

The nanoindentation tests results for samples with higher crystal phase contents are shown in Figure 14, Note that the SEM image of the glass ceramics in (Figure 10) showed that these samples are partially crystallized, that is, they contain a residual glassy phase. The calculated hardness values of, glass-ceramic materials are presented in figure 14, theincrease in the heat treatment temperature of the glass ceramics provides the highest hardness values. In the case of the Glass Ceramics, the hardness values of the Glass Ceramics heat treated at 750°C with the same 5% Eggshell content are similar and in any case higher than the glass ceramics materials that was heat treated at 650°C, probably due to the viscous flow formation as reported by the SEM image resolution in Figure 12, to cause an increase of the local strength of the Glass Ceramics, The slight differences encountered in temperature changes may outcome from their difference microstructures and the crystalline phases present. Large crystal size and intertwined structures lead to high hardness values.

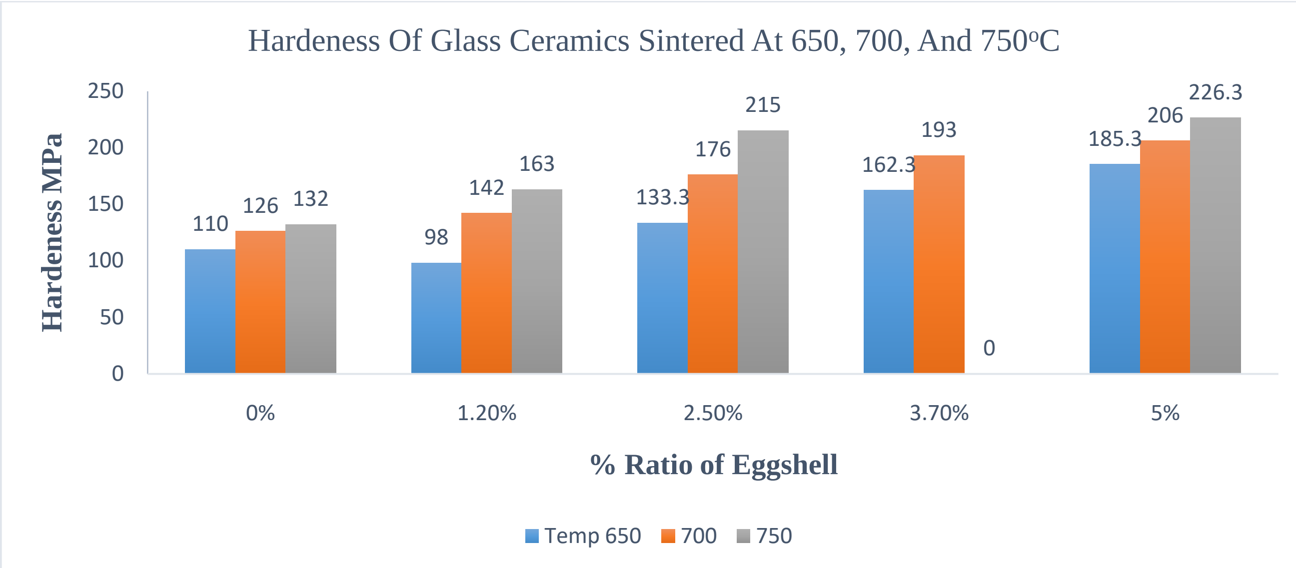


Figure 14: Hardness of the Sintered Glass Ceramics

3.6. Chemical durability

In these chemical resistance experiments, 14 compacted sample units with sizes of 25 mm diameter by 11 mm thickness were treated by immersing them in a 1M HCl solution and 1M NaOH solution for 24 hours with a drying process at 130ºCfor 4h. After washing and drying, the samples were weighed and the percentages of weight loss were calculated by taking the initial mass (m_o) and final mass (m_f) of each of the materials. It was determined using an analytical digital weighing balance with a measurement error of ± 0.0001g. The mass loss obtained was insignificance after carrying out the previous process to 14 samples, which shows the durability of the sintered glass ceramic samples at 650, 700 and 750ºCfor 1 hour; this correlates with the volume of the crystalline phases and shows excellent chemical resistance behavior for both samples. The achievement of a high chemical durability in glass ceramics indicates that the chemical composition of the crystalline phase's constituents, composition and amount of residual glassy matrix favors a good stability. Generally, glass ceramic materials have good chemical stability and often compare favorably with other ceramic type materials. An increase in the content of the crystalline phase results in greater chemical resistance in glass ceramic materials. The obtained result were in agreement with Davalos *et al*., (2020) who obtained 0.540, as the highest mass loss for chemical durability index.

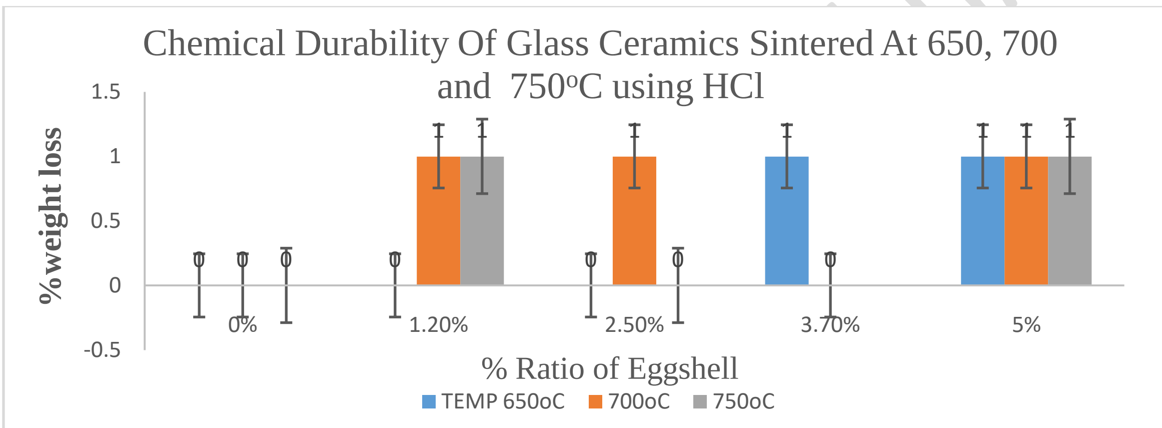


Figure 15: Chemical Durability of Sintered Glass Ceramics using HCl

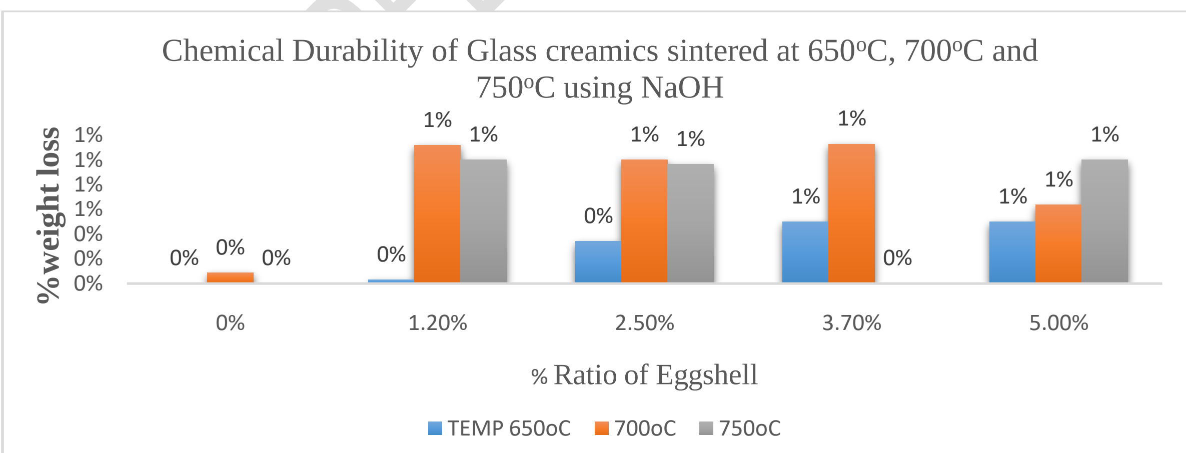


Figure 16: Chemical Durability of the sintered Glass ceramics in NaOH solution

3.7. Density of the glass ceramics

The averages density values obtained from five samples of each material. In samples GC650°C and GC700°C, the values of the density are higher in comparison to the sample GC750°C, which can be correlated with a decreased content of crystalline phases in the glass ceramics samples according to the data obtained from the Degree of crystallinity. The decrease in densityvalues fromeach sample could be correlated with the amount of orthoclase phase present in the material (GC605°C: 19.3%, GC700°C: 33%, and GC750°C: 27%), therefore, it is expected that the density increases or decreases could be as a result of increase or decrease in the crystallinity content. The obtained result is in line with the findings of Valderrama *et al*., (2021) who conducted a research on Glass-Ceramic Materials Obtained by Sintering of Vitreous Powders from Industrial Waste: Production and Properties.

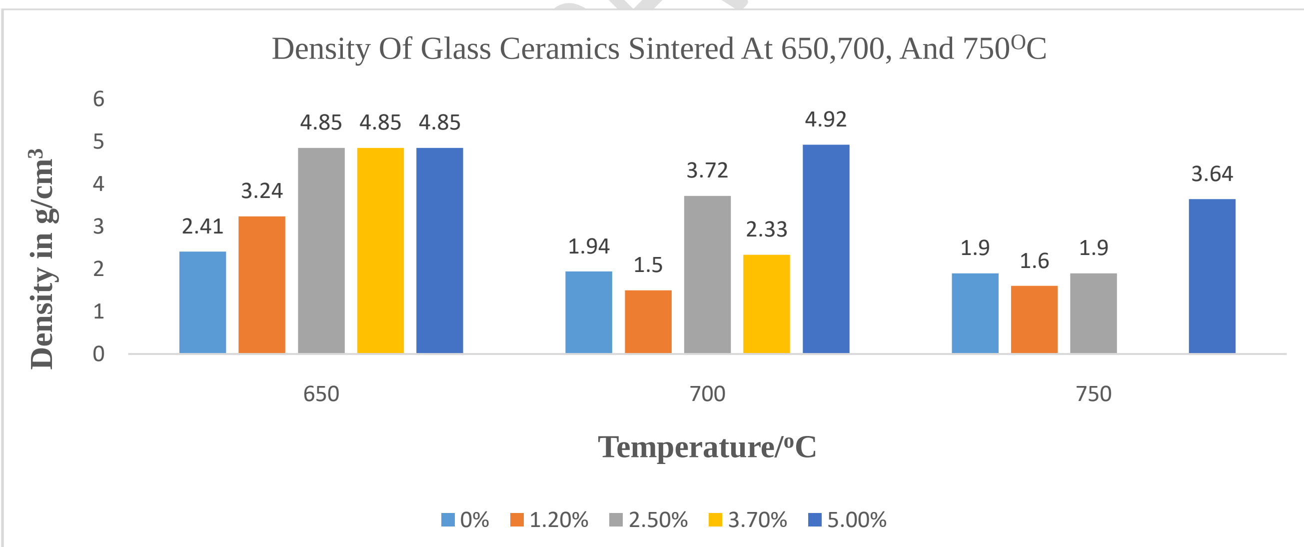


Figure 17: Density of the sintered glass ceramics

3.8.Abrasion Resistance

Figure 18present the abrasion resistance test result. It was observed that sample sintered at 700°C account for higher resistance to scratching and the attributing factor is that during sinter crystallization process, the glassy phase partially crystallizes, forming a network of interlocking crystals within the glass matrices. These crystals can be very hard and have high scratch resistance, contributing significantly to the overall abrasion resistance of the material. The size of these crystals can also play a role. Generally, finer, more uniformly sized crystals can lead to better abrasion resistance compared to larger, more unevenly sized crystals.The integration of high-scratch, hard crystals comprising both glassy and crystalline phases contribute to this resistance. The size of crystals influences their resistance, with finer and uniformly sized crystals demonstrating superior durability. Abrasion resistance correlates directly with density and compressive strength.

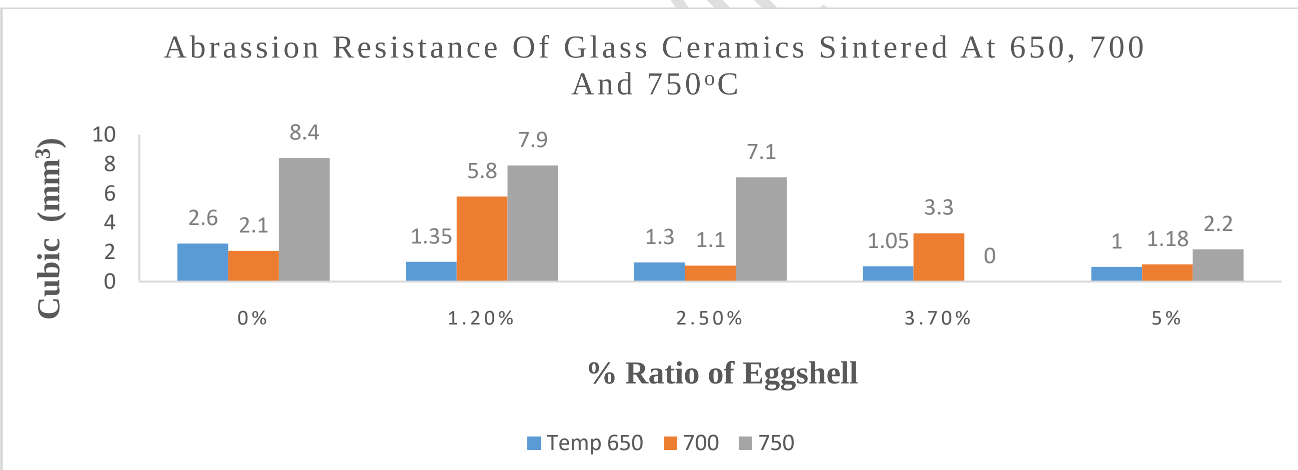


Figure 18: Abrasion Resistance of the Sintered Glass ceramics

4.1. Conclusions

- Waste soda lime silicate glass and eggshell derived CaO were successfully utilized to produce glass ceramic materials via the sinter crystallization technique, demonstrating an effective and sustainable waste valorization route.

- Chemical and phase analyses confirmed that silica rich waste glass provided the vitrifying network, while eggshell derived CaO acted as an efficient network modifier that promoted controlled crystallization and microstructural refinement.
- XRD results revealed temperaturedependent phase evolution, with quartz and calcite as the dominant crystalline phases embedded in a residual glassy matrix.
- Increasing sintering temperature enhanced densification and crystallinity up to 700 °C however, excessive heating at 750 °C caused microstructural distortion, increased pore size, and reduced mechanical performance.
- SEM observations showed improved particle bonding, reduced pore size, and a more homogeneous microstructure at intermediate sintering temperatures.
- The physical, chemical, and mechanical properties of the produced glass ceramics were found to be excellent, therefore, the glass ceramics can be used in tilling application base on the specification provide by the (ASTM C 373 standard that the ceramics tiles should have a minimum water absorption3% < E ≤ 10%, and excellent chemical durability.
- The optimum overall performance was achieved at a sintering temperature of 700 °C, which exhibited the highest mechanical strength, density, and abrasion resistance.
- The findings establish eggshell derived CaO as a viable alternative calcium source and confirm the suitability of the produced glass ceramics for tile and construction applications, supporting circular economy and sustainable materials development.

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