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Optimizing the Production of Glass Ceramic from East Lampung Basalt Rock Using Taguchi and ANOVA Analysis Methods

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Abstract

Glass-ceramics are fine-grained polycrystalline materials produced through controlled crystallization in the glass phase. The raw material for glass ceramics is basalt rock from East Lampung mixed with additives (SiO_2, MgO, and CaO). The production of glass ceramics begins with heating at a temperature of 1200 °C for 2 hours until it melts. The molten basalt rock is cooled using distilled water with a pH of 7 to produce glass material. The glass material is ground using a ceramic ball mill and filtered with a 325-mesh filter. The glass material undergoes nucleation at a temperature of 600 °C for 2 hours, followed by crystallization at 1,050 °C for 3 hours, and thermal crystallization (devitrification) at 1,050 °C for 3 hours to form a crystalline phase. The purpose of this study is to determine the optimal additive composition for the production of glass ceramics in density testing. Density testing was carried out using Archimedes' principle. The Archimedes density test is the mass of glass ceramics divided by the volume of water when the glass ceramics are placed in water. The density test results showed values ranging from 2.1 to 3.8 g/cm³, depending on the additive ratio used. An increase in CaO and MgO content tends to increase density due to the formation of crystalline phases such as pyroxene, anorthite, and olivine. Conversely, excessive addition of SiO_2 can reduce density due to the growth of amorphous phases. Taguchi analysis identified silica (SiO_2) as the number one parameter affecting density. Meanwhile, ANOVA analysis resulted in an optimal silica percentage contribution of 34.57%.

Keywords: *Glass ceramics density, basalt rock, additives, Taguchi method, ANOVA analysis.*

1. Introduction

Glass-Ceramics are ceramic materials produced through a controlled crystallization process (nucleation and crystal growth) from a parent glass [1]. Glass-ceramics possess several outstanding characteristics compared to traditional ceramics and glass, such as wear and corrosion resistance, making them ideal as abrasion-resistant coatings [2]. One potential candidate for the base material is basalt. Basalt is a gray to black, fine-grained volcanic rock that is rich in silicate and ferromagnesian minerals, such as CaO and MgO. Basalt rock, as a material, has essential quality properties, including high abrasion resistance, compressive strength, and resistance to chemical reactions. The chemical composition of basalt supports the crystallization process, resulting in crystalline phases such as anorthite, olivine, and pyroxene [3].

In Indonesia, particularly in East Lampung, basalt is abundantly available but has not been fully utilized, primarily being used as aggregate and building ornaments [4]. One region rich in basalt is the Sukadana area in East Lampung Regency. Basalt can be applied as a base material for the production of glass-ceramics [5]. Previous studies have shown that basalt from this region contains approximately 48% SiO_2, 18% Al_2O_3, 12% Fe_2O_3, 9% CaO, and 4% MgO, all of which support the formation of glass-ceramics at relatively low temperatures through a melting process at 1200–1250°C and thermal crystallization at 1050°C [6].

The production process of glass-ceramics using the devitrification heat treatment method is a key step in the manufacturing of glass-ceramics. Before creating glass-ceramics, basalt is first heated in a muffle furnace until it melts at a temperature of

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1200°C and is held for 2 hours, followed by quenching in distilled water (pH 7). Devitrification occurs when basalt is heated at 600°C for 2 hours (nucleation process), where crystals are formed from the amorphous phase [7][8]. During the nucleation stage, the precise temperature and time conditions are required to trigger the formation of crystal nuclei, while the crystal growth stage involves increasing the size of the crystals in the glass matrix. This process can be controlled to achieve the desired crystal size and distribution, which in turn will enhance the mechanical and thermal properties of the resulting glass-ceramics [9]. After being held for 1 hour, the temperature is increased to 1050°C and maintained for 3 hours. At this temperature, crystallization occurs, and the glass-ceramics can be used.

The addition of additives such as SiO_2, MgO, and CaO to basalt has the potential to modify the microstructure and density of the resulting glass-ceramics. Several studies have shown that increasing the CaO and MgO content tends to promote the growth of crystalline phases (such as pyroxene and anorthite), which significantly enhances the density and hardness of the material [10]. On the other hand, excessive ad dition of SiO_2 can increase the formation of the amorphous phase, which may reduce the final density of the material [1], [11]. The density of basalt-based glass-ceramics generally ranges from 1.9 to 2.9 g/cm^3, depending on the composition and processing conditions applied.

To optimize the utilization of basalt from East Lampung, it is necessary to engineer the chemical composition through the variation of additives to control the density. The Taguchi analysis method can be applied in this study to determine the ranking of additives (SiO_2, MgO, and CaO) that most influence the material's density. Through this analysis, it is expected to obtain an optimal formula to produce glass-ceramics with properties suitable for specific needs and to support the development of advanced materials based on Indonesia’s natural resources [12][13].

2. Methods

The design of experiment (DoE) is shown in Table 1.

Table 1. Sample numbering and wt % of additives

No. Sample	Additive materials (wt %)		
	SiO_2	MgO	CaO
1	10	5	7
2	10	8	10
3	20	5	10
4	10	8	7
5	10	5	10
6	20	8	10
7	20	5	7
8	20	8	7
9	none	none	none

The manufacture of basalt glass ceramics begins with refining the basalt using a ball mill followed by sifting the 325 mesh. The basalt powder is then melted using a muffle furnace at a temperature of 1250 ^oC for 2hrs. It was quenched with pH 7 water at room temperature. The nucleation temperature was kept at 600 ^oC for 1hr and the crystallization temperature at 1.050 ^oC for 3hrs.

The Taguchi method uses matrix design methodologies such as 2x2 or 3x3 to minimize experimental effort. For example, a 3x3 design yields 9 efficient experimental parameters that can be used. The Taguchi method can analyze how initial parameter variations affect the characteristics of the test results (response). The Taguchi method uses the signal-to-noise ratio (S/N) as a quality indicator [14].

Taguchi method with orthogonal arrays (OA). Orthogonal arrays (OA) use individual factors (e.g., additives of SiO_2, MgO, and CaO) and their interactions with key response variables (i.e., material density), while keeping the number of experiments under control [15][16]. In this study, the orthogonal matrix varied the composition of three main silica additives (SiO_2), magnesium (MgO), and calcium (CaO) in the production of basalt-based glass ceramics. After 9 glass ceramic samples were made using the Taguchi method, density testing was carried out. Density testing of Glass Ceramics with additives using Archimedes' law:

$$\rho = \frac{m}{V} \tag{1}$$

where: ρ: density; m: mass; and V: Volume

By analyzing the influence of these additives on the resulting material density, this method can rank their effects and identify the most significant composition influencing density. This study employs Analysis of Variance (ANOVA). The ANOVA method is used to assess the percentage contribution of production parameters (additives) to the density test results [17]. The ANOVA method presents the error values obtained in this study. Acceptable error values in research are typically less than 0.05%. Error values exceeding 0.05% are caused by other parameters influencing density.

3. Result and Discussion

This study discusses the results of glass-ceramic density data. Density testing was conducted using Archimedes' principle. Density results were analyzed using Taguchi and Analysis of Variance (ANOVA).

3.1 Density Testing Results

Density testing of Glass Ceramics with additives using Archimedes' law. Archimedes' principle is that the mass of a material is divided by the increase in volume when the material enters water. The density results are shown in Figure 1.

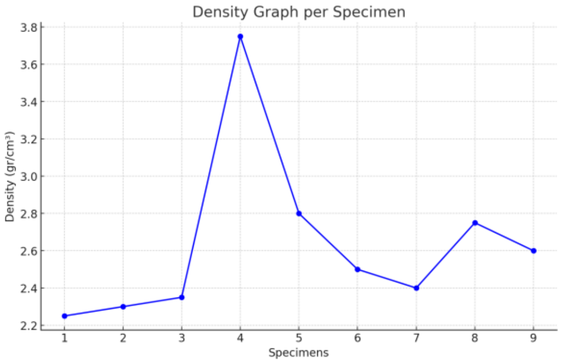

Figure 1. Density Testing Results

The density profile of the nine basalt-derived glass-ceramic specimens reveals a nuanced interplay between SiO_2, MgO and CaO contents (Table 1). At constant SiO_2 of 10 wt%, raising MgO from 5 wt% to 8 wt% and CaO from 7 wt % to 10 wt% (from specimens 1 to 2) does not affect the density (both 2.35 g cm^{-3}). This corroborates Aliyu’s assertion that a relatively high silica fraction dominates the structural network, limiting the effectiveness of additional divalent oxides in promoting compact crystallization [18].

Increasing SiO_2 to 20 wt% while holding MgO and CaO at 5 wt% and 10 wt%, respectively (Specimen 3), lowers the density to 2.28 g cm^{-3}. Elevated silica enhances melt viscosity, favours glassy residuals and suppresses crystal nucleation, thus enlarging the amorphous phase and diminishing bulk compaction behaviour previously observed by [19].

Conversely, Specimens 4 and 5 yield the highest densities, 3.81 g cm^{-3} and 3.70 g cm^{-3}, with only 10 wt% SiO_2 but increased MgO (8 wt%) or CaO (10 wt%). Here, MgO acts as a potent crystal-field modifier and nucleating agent, while CaO stabilises high-density crystalline phases (e.g., anorthite). The synergistic balance between a low-viscosity melt (low SiO_2) and an optimal divalent-oxide population fosters rapid crystal growth and pore annihilation, explaining the marked densification.

Specimen 6 (20 wt% SiO_2, 8 wt% MgO, 10 wt% CaO) drops sharply to 2.40 g cm^{-3} despite ample MgO and CaO, underscoring that silica content governs glass–crystal competition. Similar silica-rich systems require additional nucleators (e.g., TiO_2) to offset this effect, as reported by [20].

The lowest density, 2.11 g cm^{-3} (Specimen 8), arises from the combination of high SiO_2 (20 wt%) with asymmetric MgO/CaO (8/7 wt%). Here, excess MgO in a viscous silica matrix promotes heterogeneous phase separation rather than coherent crystallisation, leading to residual porosity. Finally, Specimen 7 (20 wt% SiO_2, 5 wt% MgO, 7 wt% CaO) recovers marginally to 2.48 g cm^{-3}, indicating that reducing MgO moderates the viscosity mismatch and partially restores densification.

Collectively, these results demonstrate that silica level is the primary lever controlling densification, while MgO and CaO exert secondary, but synergistic, influences. Optimal densities (> 3.7 g cm^{-3}) are achieved when SiO_2 is limited to about 10 wt% and the MgO/CaO ratio is tuned to maximise crystal nucleation without impeding melt flow. The present findings refine existing models of glass-ceramic crystallisation and provide a compositional roadmap for engineering basalt-based glass-ceramics with superior mechanical integrity and potentially enhanced functional properties.

3.2 Taguchi Analysis for the Density of Glass Ceramics Product

The Taguchi method helps in identifying and ranking parameters based on their influence, providing valuable insights for designers and engineers in customizing and improving production processes [21][22]. The results of the Taguchi analysis are shown in Table 2 below.

Table 2. Taguchi Analysis Results

Level	SiO	MgO	CaO
1	3.053	2.703	2.688
2	2.317	2.667	2.683
Delta	0.735	0.035	0.005
Rank	1	2	3

The Taguchi response-mean analysis unequivocally identifies SiO_2 as the primary determinant of bulk density in basalt-derived glass-ceramics. At 10 wt % SiO_2 (Level 1) the mean density reaches 3.053 g cm^{-3}, whereas doubling the silica to 20 wt % (Level 2) lowers the mean to 2.317 g cm^{-3}, yielding the largest Δ-value (0.735). This steep decline highlights the sensitivity of the system to silica content and underscores the need for stringent control of network-forming oxides during composition design [23].

The reduction in density with rising SiO_2 is consistent with the amorphous-phase hypothesis, whereby excess silica elevates melt viscosity, suppresses nucleation and ultimately favours a glassy residual that is intrinsically less dense than its crystalline counterpart [23]. Consequently, lowering SiO_2 is an effective strategy to promote crystallisation-controlled densification and to achieve superior mechanical integrity.

MgO exerts a secondary, yet non-negligible, influence: increasing its level from 5 wt % to 8 wt % reduces the average density only marginally (from 2.703 to 2.667 g cm^{-3}; $\Delta = 0.035$). Although MgO is widely acknowledged as a nucleating agent, its impact proves composition-dependent; the present data suggest that its nucleation efficacy is moderated by the prevailing SiO_2 concentration and the accompanying CaO fraction, corroborating observations.

By contrast, CaO displays the least influence within the tested window. A shift from 7 wt % to 10 wt % changes the mean density by just 0.005 g cm^{-3} (from 2.688 to 2.683 g cm^{-3}), indicating an almost negligible main-effect contribution. While CaO is known to stabilize crystalline phases such as anorthite, the narrow variation applied here appears insufficient to elicit a measurable density response.

The Taguchi framework confirms that reducing SiO_2 is the most effective route to maximise densification, with MgO offering a moderate benefit and CaO imparting only minor changes under the examined conditions. These insights provide a quantitative hierarchy of compositional levers for designing high-density basalt glass-ceramics and lay a solid foundation for subsequent optimisation studies aimed at enhancing their mechanical performance. In the Taguchi analysis, a main effects plot for means was obtained. The results of the main effects plot for means are shown in figure 2.

The main-effects plot for mean density offers a succinct quantitative perspective on how each oxide – SiO_2, MgO and CaO – governs bulk densification in basalt-derived glass-ceramics. A steep negative slope is evident between 10 wt % and 20 wt % SiO_2: the mean density falls from about 3.1 g cm^{-3} to about 2.3 g cm^{-3}. This trend confirms that increasing silica promotes a larger residual glassy network, elevates melt viscosity and suppresses crystal growth, ultimately reducing packing efficiency. The observation is fully consistent with the glass-network former model and with data reported for related silicate systems [24].

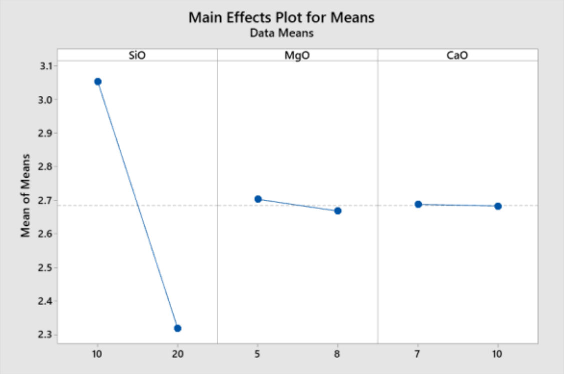

Figure 2. Main effects plot graph for the mean

MgO (magnesia). The MgO trace is nearly horizontal; raising MgO from 5 wt % to 8 wt % lowers the mean density only marginally (from about 2.71 to 2.67 g cm^{-3}). Although MgO is recognised as a crystal nucleator, its isolated main effect is modest within the studied window. The implication is that MgO's ability to enhance densification is highly contingent upon its interplay with SiO_2 and CaO, rather than on its concentration alone a point that warrants targeted follow-up experiments.

The CaO line is virtually flat, with mean densities of ≈ 2.688 g/cm^3 at 7 wt % and about 2.683 g/cm^3 at 10 wt %. Such negligible variation indicates that, under the present compositional constraints, CaO neither disrupts nor appreciably enhances the densification pathway, a finding that echoes the limited main-effect influence documented by [25]. Collectively, the plot ranks SiO_2 as the dominant density-controlling factor, MgO as a secondary modifier and CaO as essentially neutral. These insights reinforce the strategic imperative of limiting silica content while judiciously balancing divalent modifiers to achieve high-density glass-ceramics with favourable mechanical properties, validating the efficacy of the Taguchi methodology for compositional optimisation.

3.3 ANOVA Analysis of the Glass Ceramics Density Produced with Additives

ANOVA is a statistical analysis method that belongs to the branch of inferential statistics. Statistics is a branch of science that studies how to collect, analyze, and interpret data. The ANOVA data produced are DF, Adj SS, Adj Ms, Seq SS, Contribution, and P-value. The ANOVA results are shown in Table 3.

Table 3. Taguchi Analysis Results

Source	**DF**	**Seq SS**	**Contribution**	**Adj SS**	**Adj MS**	**F-Value**	**P-Value**
SiO_2	1	0.55942	34.57%	0.55942	0.55942	2.20	0.212
MgO	1	0.03282	2.03%	0.03282	0.03282	0.13	0.737
CaO	1	0.01002	0.62%	0.01002	0.01002	0.04	0.852
Error	4	1.01576	62.78%	1.01576	0.25394		
Total	7	1.61802	100.00%				

The results of the ANOVA analysis show the influence of three factors, namely SiO_2, MgO, and CaO, on density. Based on the P-Value, none of these factors have a significant effect on density. The P-Value for SiO_2 is 0.212, which is greater than 0.05, indicating insufficient evidence to claim that SiO_2 significantly affects density. The P-Value for MgO is 0.737, and for CaO, it is 0.852, both of which are also greater than 0.05, suggesting that these two factors do not have a significant impact on density. However, in terms of contribution, SiO_2 accounts for the largest portion of the variability in density, with a contribution of 34.57%, despite its lack of significant effect. On the other hand, MgO and CaO contribute much less, at 2.03% and 0.62%, respectively. Overall, although SiO_2 provides the largest contribution to the variability in density, none of the factors exhibit a significant influence on density at the 95% confidence level.

Other factors influencing density include sintering temperature, material composition, and degree of crystallization. In the study by Ayoob et al. (2011) [26] it was shown that increasing the sintering temperature can increase density and improve the mechanical properties of glass ceramics, even surpassing those of traditional ceramics. However, in the study by Kumar et al. (2009) [27] it was found that glass compositions with more than 70% content can reduce densification at higher sintering temperatures. In addition to temperature, material composition also plays a significant role in determining the density of ceramic glass. In the study by Aliyu (2020) [18] it was found that increasing the crystalline phase content in ceramic glass can increase density. Meanwhile, Zhang & Gao (2008) [28] found that adding more than 50% glass components to the base material can reduce relative density due to porosity caused by grain growth. Additionally, sintering processes and crystallization control contribute to density variations. Effective crystallization control can reduce surface topography damage caused by pores formed during heat treatment. Glass ceramics with a crystallization degree exceeding 50% can enhance material density and strength [29][30].

4. Conclusion

This study aims to investigate the effect of SiO_2, MgO, and CaO content on the production of basalt-based glass-ceramics for density testing. The results show that SiO_2 plays a dominant role in influencing the density process. A decrease in SiO_2 content from 20% by weight to 10% by weight significantly increased the average density from approximately 2.3 g/cm^3 to 3.1 g/cm^3. This behavior is associated with the role of SiO_2 as a strong glass network former, which at higher concentrations increases viscosity and promotes the formation of an amorphous phase, thereby inhibiting crystal growth. On the other hand, MgO showed a moderate effect, possibly due to its role as a nucleating agent, while CaO had a minimal impact on density in the tested range. These findings are in line with the research objective, confirming that composition adjustment, especially SiO_2 content, is critical for optimizing glass ceramics production for density and glass-ceramic microstructure compactness. The results of the ANOVA analysis show that SiO_2, MgO, and CaO do not significantly affect the production of glass ceramics for material density, as their respective P values (0.212, 0.737, and 0.852) are all greater than 0.05, indicating insufficient evidence for a significant impact. Nevertheless, SiO_2 contributed the most (34.57%) to the variability of glass ceramics production for density, while MgO and CaO contributed much less, at 2.03% and 0.62%, respectively.

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