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The high-hardness ceramic glazes based on basalt from Bali Province for ceramic body coatings

Komang Nelly Sundari [1], Subari [1], David C Birawidha [2], Hendra Prasetia [2], I Wayan Mudra[3], Yusup Hendronursito [2]

[1] Research Center for Advance Material, National Research and Innovation Agency, PRMM, BRIN, Banten 15314, Indonesia

[2] Research Center for Mining Technology, National Research and Innovation Agency, PRTPB, BRIN, Tanjung Bintang 35361, Indonesia

[3] Arts Crafts Study Program, Faculty of Fine Arts and Design, Indonesian Art Institute of Denpasar, Bali 80235, Indonesia

Corresponding author: yusu012@brin.go.id (Y. Hendronursito)

Abstract: The ceramic glaze method is not only done to add color aesthetics but also to increase the hardness of the glaze. Basalt is one of the raw materials developed as a ceramic glaze material because it is cheap, easy to find, and has good characteristics. This research aims to determine glaze characteristics by varying the concentration of basalt rocks from the Ababi area, Karangasem Regency, Bali Province. The experiment used basalt at 45%-61%, Feldspar 27%-37%, Kaolin 3%-8%, and ZnO 7%-10% as raw materials for glaze, not frit glaze. Heating the glaze material layer at 1,250 °C for 3 hrs. The product characteristics of glaze ceramics include chemical composition, crystal phase, microhardness, porosity, density, thermal expansion coefficient, and surface morphology. Basalt from Bali is characterized by its high Fe_2O_3 content, reaching 20.07 wt%. Compared to basalt in generally, it has a different impact on the physical and mechanical characteristics of the ceramic glaze produced. Based on the observation of ceramic glaze products, the glaze composition with 56% basalt, 32% feldspar, 5% kaolin, and 7% ZnO showed the best coating, with a yellowish-brown color and an average thickness of 79.82μm. The hardness value of the glaze layer is 6.5 GPa, exceeding the standard hardness value of glazes on the market. Glazed ceramics contain the minerals Gahnite ($ZnAl_2O_4$) and Coesite (SiO_2), which can increase the hardness of the glaze. This research demonstrates the tremendous potential and added value of using basalt from Bali Province as a raw material for glazed ceramics.

Keywords: glaze, ceramic, basalt, hardness, gahnite, coesite

1. Introduction

A glaze is a liquid suspension with small amounts of mineral granules that is applied by pouring, brushing, dipping, or spraying onto the surface of a ceramic biscuit. After drying, it is fired again at a temperature where the contents inside will melt to form a layer of glass whose surface is coated with glaze material (Brian and Carleton, 1982; Quang, 2023).

Based on the raw material, glaze can be divided into raw and frit. The raw glaze is a material that uses a mixture of raw materials that can be directly glazed on the ceramic body. The glazed ceramic body can be fired once or twice. This glaze is usually applied to stoneware bodies, requiring firing temperatures between 1,150 to 1,300°C. (Yeşilay, 2019). At the same time, frit glaze is a glaze whose raw material is burned or melted first before being used as a raw material for making glaze. This frit glaze is used at temperatures below 1,100 °C (Yeşilay, 2019). Decorated glaze materials can be divided into four components: building blocks, melting agents, thickeners, and colorants. The color of the glaze depends on the metal oxide in it; for example, the glaze will be brown if it contains iron (Fe_2O_3), green if it contains copper (CuO) or chrome (Cr_2O_3), and blue if it contains cobalt oxide (Co_2O_3) (Yeşilay, 2019).

Ceramic items covered with glaze have a high aesthetic value. The beauty of the glaze is influenced by three factors: the suitability of the glaze material to the ceramic body, the surface layer of the glaze,

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and the color of the glaze itself. In addition to an attractive and shiny appearance, the hardness of the glaze is a determining factor for glaze for ceramic coatings (Bao et al., 2024; Prstić et al., 2007).

The most common raw materials for making glaze are feldspar and quartz. Both types of materials are found in volcanic rocks that result from volcanic eruptions. These parent rocks will fuse into other minerals, such as kaolin and quartz. Previous researchers have tried volcanic rocks such as andesite, perlite and basalt as raw materials for glazes, as they contain high amounts of iron oxides and alkalis (Ergul et al., 2007; Sariisik, 2021; Simic and Gilic, 2000). The potential of basalt rock as a glaze material is enormous because, besides having aesthetic value, basalt as the main component of the glaze mixture can improve mechanical properties, chemical resistance, and friction (Andrić et al., 2012; Bayrak and Yilmaz, 2014; Gultekin E. E, 2018). Basalt rocks in Indonesia are scattered in various regions, such as Sukadana basalt from Lampung province (Birawidha et al., 2023; Isnugroho et al., 2020). Meanwhile, basalt rocks in Bali Province can be found in the Mount Agung area in the Ababi area, Karangasem Regency, Bali Province. Currently, basalt rocks from the Ababi area are used as ornaments or statues with their characteristics. Conversely, the rock powder is just waste that has yet to be utilized optimally, especially as a glaze material.

This research explores the potential utilization of basalt rock from Bali Province as a glaze material with unique color characteristics and high hardness. Basalt-based glaze products are expected to provide added value to local materials as advanced materials and support the surrounding community's economy.

2. Materials and methods

The raw materials for the production of the glaze consist of basalt rock from the Ababi area, Karangasem Regency, Bali Province, Indonesia, China feldspar, kaolin from Bangka - Indonesia, and zinc oxide (ZnO). The feldspar used in this research comes from Chinese products with a size of 200 mesh. Feldspar is a silicate mineral rich in potassium; in this feldspar, there is 13% K2O, and it is widely used as a ceramic material. The composition of feldspar and caolin shown in Table 1.

Table 1. Chemical composition of materials

Compound	Felsdpar (wt%)	Caolin (wt%)
SiO_2	66	48.082
Al_2O_3	18	36.129
Fe_2O_3	0.05	0.648
CaO	0.04	0.014
K_2O	13	0.329
TiO_2		0.256
P_2O_5		0.858
Na_2O	10	0.041
MgO	0.04	0.016

The production of glaze from basalt rock refers to the research of Prstic A. et al. 2005 (Prstic et al., 2003). However, in this study, boric acid and opalite minerals were not used because these ingredients are only used for low-temperature glaze formulas. In contrast, the stoneware bodies studied have a mature body temperature of about 1,250 °C (Brian and Carleton, 1982). However, the use of Ababi basalt rock in Karangasem Regency increases to above 40 to 61%, which is a raw glaze, not a frit glaze. The composition of the glaze raw materials is shown in Table 2. This composition referred to the results of previous research by Prstic A et al. 2003 as well as mineral content that can be used as raw material for the glaze (Prstić et al., 2003).

The glaze production begins by grinding the basalt rock until a size of 325 mesh is obtained, then stirring using a mortar grinder and adding enough water to make it homogeneous with the ratio according to Table 2. Try the glaze on a test piece of stoneware mass. Next, burn the test object at a temperature of 1,250^{o}C.

Table 2. The Composition of experiment

Material/ Samples code	GB 8	GB 15	GB 18
Basalt powder (wt %)	61	45	56
Feldspar (wt %)	27	37	32
Kaolin (wt %)	3	8	5
ZnO (wt %)	9	10	7

The characterization of basalt from Bali was carried out by testing chemical composition using Panalytical Xpert³ Powder XRF Spectrometer from Malvern Panalytical operating at 50 kV and 3 mA. The crystalline phase was measured in powdered basalt state under 325 mesh using X-ray Diffraction (XRD) Merck Panalytical X'pert³ Powder XRD at 40 kV and 30 mA, in the interval 5-80 degrees 2θ reflections. XRD data analysis by HighScore software offers X-ray diffraction data processing, phase identification, reporting and profile suitability. The glaze morphology using 3 mA and FE-SEM Thermo Scientific Quattro S.

The glaze product color testing uses a colorimeter Merck CHN-Spec Model CS10 color reader. This tool works to determine color based on the blue, red and green color components of the light absorbed by the glaze sample. This color meter works based on the Beer-Lambert law, which states that the absorption of light transmitted through a medium is directly proportional to the concentration of the medium (Shrestha and Shrestha, 2016).

The method of coefficient of thermal expansion (COE) analysis involves using a one-liter dilatometer flask with an attached capillary-bulb arrangement with electrical contacts spaced over a calibrated volume. The flask is filled with aggregate and water and placed in a bath that automatically heats and cools to maintain equilibrium at different electrical contacts. The equilibrium temperature is measured with a Beckmann thermometer. The process is repeated at another contact. This apparatus determines the cubical thermal coefficient of expansion, from which the mean linear thermal expansion can be calculated. The measurement range is approximately 7°F, but the temperature increment and base temperature can be adjusted. The accuracy of the apparatus is confirmed by comparing results with known materials. The thermal coefficients of various fine and coarse aggregates have been measured, with mean linear thermal coefficients ranging from 3.0×10^{-6} to 7.1×10^{-6} per °F.

A Zwick Roel tool performed the micro-Vickers test with a 0.1N load for 12 scond. The indentations were inspected using differential interference optical microscopy. An NIH-Image computer program was utilized to measure the diagonal size of digital micrographs obtained after every indentation. To calculate microhardness Vickers (HV), utilize the subsequent equation 1 (Sukmana et al., 2023):

$$H_V = 1.8544 \frac{P}{d^2} \tag{1}$$

where P is the indentation load (0.1N), d is the indentation diagonal (mm).

Physical properties were seen from testing the coefficient of thermal expansion (COE), porosity and density based on ASTM C373-88. The porosity and density values of the samples follow the standard (ASTM C373-88, 2006), using equations 2 and 3.

$$\rho = \frac{m}{v} \tag{2}$$

where: ρ is the density (g/cm3), m is the sample mass (g) and v is the sample volume (cm^3).

$$Porosity\ (\%) = \frac{m_b - m_k}{\rho_{water}\ x\ V_t}\ x\ 100\% \tag{3}$$

where: m_b is the mass of the wet sample (g); m_k is the mass of the dry sample (g), ρwater is the density of water (g/cm^3), and V_t is the volume of the sample (cm^3).

3. Results and discussion

A real example of applying glaze made from Ababi area – Bali Province basalt rock to a ceramic bowl is shown in Fig. 1.

3.1. Chemical composition of basalt rocks from Bali province – Indonesia

The chemical composition of Ababi basalt rock, Bali is shown in Table 3The chemical composition of

Fig. 1. The application of glaze ceramics based on basalt from the Bali Province

the raw material dramatically influences the characteristics of the final glaze product, especially surface parameters such as color, gloss or roughness (Ergin et al., 2023). Based on the results of chemical composition testing, basalt from Bali Province is dominated by 46.497% SiO_2, 20.072% Fe_2O_3, and 12.798% CaO.

Table 3. the chemical composition of Basalt from Bali Province and the comparison

Compound	Bali Basalt	Lampung Basalt (Sukmana et al., 2022)	Kaponik (Serbia) Basalt (Prstić et al., 2007)	Turkey (Sariisik, 2021)
	Chemical Composition (wt%)			
SiO_2	46.497	48.418	48.920	49.47
Al_2O_3	14.721	18.820	19.990	17.24
Fe_2O_3	20.072	12.595	7.910	10.26
CaO	12.798	9.761	8.390	10.35
K_2O	2.588	0.636	2.390	0.61
TiO_2	1.549	1.329		1.33
MnO	0.354	0.194		0.16
P_2O_5	0.859			0.22
SrO	0.147			
Na_2O		3.356	2.890	3.42
MgO		4.561	6.380	6.24
SO_3				0,07
Total	99.585	99.670	96.870	
Ignition loss	0.415	0.330	3.130	0.53

Based on previous studies that utilize basalt rock as ceramic material, there are some differences in chemical composition. Compared to other studies that use basalt as a ceramic raw material, basalt from Bali has a lower chemical composition of SiO_2, Al_2O_3, Na_2O, and MgO. In contrast, the composition of Fe_2O_3, CaO, K_2O, TiO_2, and P_2O is higher than other basalt. The difference in the chemical composition of this basalt is expected to affect the differences in the physical and mechanical properties of the glazed ceramics produced. The high Fe_2O_3 content in basalt from Bali Province will result in the color of the glazing product. In a previous study that used Sidoarjo mud material, which mainly contains iron oxide (Fe_2O_3) and basalt rock, the color of the glaze produced ranged from greenish to brown (Supriyadi et al., 2012). Likewise, zinc oxide (ZnO) strongly affects getting a dull glaze color (matte glaze). If iron oxide (Fe_2O_3) and zinc oxide (ZnO) are used together in the glaze, the glaze color will be from pale yellow to mustard brown (Çakı et al., 2007). After reacting with ZnO, the very high levels of Fe_2O_3 in Bali basalt will likely give a dull color effect (matte glaze) when the glaze melts after being fired at high temperatures. In this study, 27% to 37% feldspar material was used. Fe_2O_3 that interacts with feldspar functions as a flux agent. High Fe_2O_3 and CaO will reduce the viscosity value of the melted basalt melt. In addition, the high presence of TiO_2, around 2%, also shows a tendency for high crystallization with a spontaneously fine crystal structure (Ergul et al., 2007).

3.2. The crystal phase of basalt from Bali Province

The results of XRD interpretation using High Score Plus (HSP) observed were orthopyroxene (COD 969010425) and plagioclase consisting of anorthite (COD 969000363) and albite (COD 969001634), shown in Fig. 2.

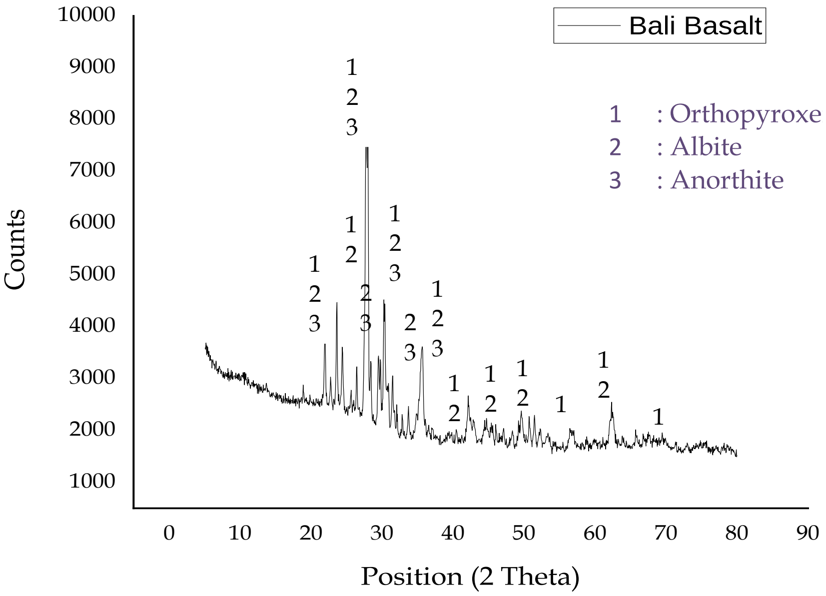

Fig. 2. Crystall phase of Basalt from Province Bali

Usually, in basalt, there are also olivine minerals (Prstić et al., 2007; Sukmana et al., 2022). The absence of olivine minerals in Ababi basalt rocks is thought to be because these rocks are basaltic magma formed under high-pressure conditions (Prstić et al., 2003). Although olivine minerals do not appear on the XRD graph, basalt rocks from Bali Province, classified as volcanic rocks, can still be used as glaze raw materials (Setiadi and Sundari, 2021). Based on the pattern list from the analysis results using Highscore plus, only the orthopyroxene phase (COD 969001751) has a chemical composition bond to Fe. In contrast, the other phases, namely albite (COD 969001634) and anorthite (COD 969000363), do not have a bond with Fe. Furthermore, the orthopyroxene phase has the general equation (Mg, Fe, Ca) (Mg, Fe, Al), (Si, Al)2O6. The natural composition dominated by Fe_2SiO_6 is ferrosilite (Haldar, 2020). According to X-ray fluorescence of basalt from Bali, the high content of Fe_2O_3, which reaches 20.072%, binds with SiO_2, which reaches 46.497% in pyroxene minerals to form orthoclase.

Anorthite (COD 969000363) is a class of minerals that create plagioclase rocks found throughout nature. The structure of Anorthite belongs to the group of SiO_4 and AlO_4 tetrahedra connected by Ca^+. Glass ceramics with an anorthite crystal phase can be used as ceramic biomaterials because they are non-toxic and harmless (Agathopoulos et al., 2003). Ceramics with Anorthite as the main crystalline phase will have moderate final product thermal expansion coefficient and low dielectric constant (Abdel-Hameed and Bakr, 2007).

3.3. Application of glaze on ceramic body

The glazing results on the ceramic body for the three sample compositions are shown in Fig. 3.

There is a significant color difference from the third sample despite the same melting temperature

Fig. 3. Glaze ceramics test material; a). GB8; b). GB15; c). GB18

of 1,250°C. The glaze has melted for glaze formulas GB8 and GB15, and the color is evenly brown. There are glaze defects such as small holes and bloating due to exceeding the glaze cooking temperature (overfiring) seen with a magnifying glass magnification of 100 times, unlike the GB18 glaze. The glaze is perfectly cooked, and on the surface of the glaze, it looks like brown glaze crystals. The resulting glaze has a faded brown: yellow (brassy brown) color. This color is due to the relatively high iron oxide (Fe_2O_3) content in the basalt rock, which is 20.072%. This color difference is caused by the presence of dark-colored minerals in the basalt rock, such as the mineral orthopyroxene with the chemical formula $(Mg, Fe)_2Si_2O_6$ in greyish white, which, if this orthopyroxene reacts with ZnO with a percentage content below 10% by weight will give a glaze color like crystal. This result is similar to previous research conducted by Tulyaganov et al., 2007 (Tulyaganov et al., 2007), making glaze for body porcelain using ZnO around 5-8%. Thus, the glaze formula code GB18 of the fired glaze shows yellowish-brown glaze crystals that fade in the glaze layer. However, in sample GB18, the evenness of the color is not good. There is also melting in the glaze layer that looks like crystals. The cohesive force causes the melting.

The phenomenon of color differences in the three samples with different compositions is shown in Fig. 4. The difference between the GB18 and the other two samples is visible. Sample GB18 looks brighter (L*) and more yellow (b*) than samples GB8 and GB15. However, these three samples tend to approach green (a*) with values that are almost close together. Samples GB8 and GB15 tend for color levels to be close together and even coincide when viewed from the color test results diagram. Based on the visual observation results, formulations code GB 8 and GB 15 have defective glaze characteristics, so the most optimal sample is GB 18, which was characterized. According to Ergin and Co-Workers (2023), the grain size of the raw material significantly influences the whiteness and gloss and reduces the surface roughness of the glaze (Ergin et al., 2023). This research did not vary the grain size or temperature, so the chemical composition of the raw material significantly influenced the color of the glaze obtained.

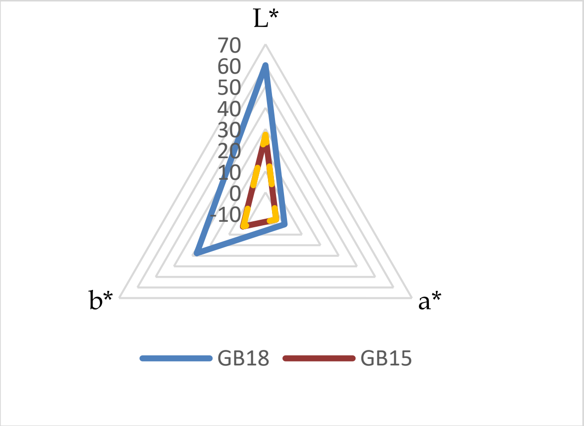

Fig. 4. Color differences based on glaze ceramics sample components

3.4. The chemical composition of glaze ceramics

The chemical composition of glaze ceramics from sample GB18 is dominated by 16.056% Al_2O_3, 49.574% SiO_2, and 6.11% Fe_2O_3, as shown in Table 4.

The addition of feldspar minerals and zinc oxide provides a significant change in the chemical composition of the glaze mixture. Iron compounds fell quite drastically to 6% from the previous 20%, replaced by an increase in zinc oxide, silica, and alumina. This change in chemical composition will affect the results of crystal diffraction that appears in the formed glaze layer, which will affect the resulting mechanical properties.

Table 4. The chemical composition of glaze ceramic sample GB18

Compound	Glaze Ceramics (GB18)
SiO_2	49.574
Al_2O_3	16.056
Fe_2O_3	6.110
CaO	5.700
K_2O	7.349
TiO_2	0.667
MnO	0.116
P_2O_5	0.625
ZnO	13.42
Rb_2O	0.116

3.5. Crystal phase of glaze ceramics

Thin-layer samples were prepared for XRD testing by cutting the glaze samples as thin as possible with a precision thin-cutting tool. The tool is set to thin layer mode to read only the scratches on the surface in the XRD tool. Based on Fig. 5, the amorphous phase dominates the crystalline phase in the formed glaze layer. The precipitation forms several crystalline phases, such as Gahnite (COD 969007028) and Coesite (COD 969000804). Gahnite has a chemical structure of $ZnAl_2O_4$ and Coesite SiO_2. With the addition of zinc oxide to the glaze composition, alumina in plagioclase tends to bind with zinc to form Gahnite, especially in the presence of TiO_2, which provides an opportunity for Gahnite formation because it can trigger the nucleation of gahnite crystals (Abo-Mosallam et al., 2013). Silica precipitation also occurs to form coesite due to the addition of feldspar to the glaze mixture. Coesite should only form under considerable pressure, but there are enough times when coesite can form from amorphous silica when heated at low temperatures (Martínez et al., 2008).

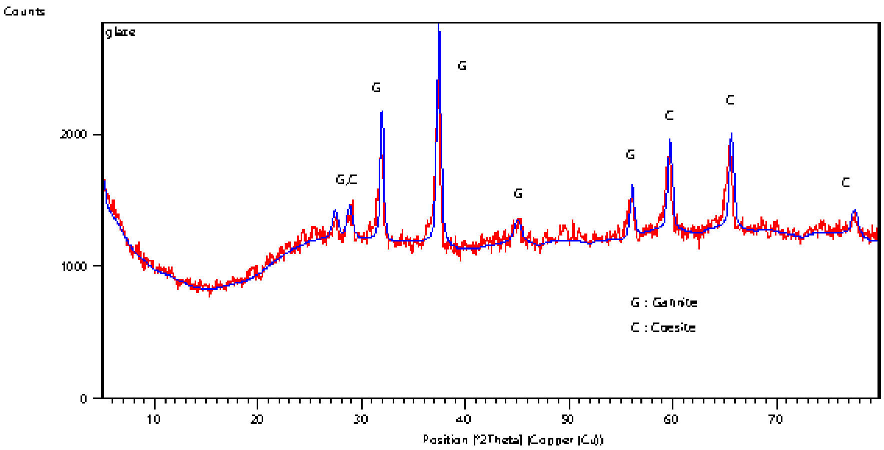

Fig. 5. Crystal phase of glaze ceramics

3.6. Thermal coefficient of glaze ceramics

The coefficient of thermal expansion (COE) test results for ceramic bodies and glaze material code GB 18 using the SNI ISO 10545-8 test method are shown in Fig. 6. it appears that the COE value for ceramic bodies (6.2 x 10 $^{-7}$/°C) is greater than that of The COE value of the glaze (3 x 10^{-7}/°C), so the glaze material can stick perfectly to the surface of the ceramic body, meaning that there is a match between the body and the glaze (body glaze fit) after being fired at a specific temperature, namely below 1,280 °C (Birawidha et al., 2023; Prstić et al., 2007).

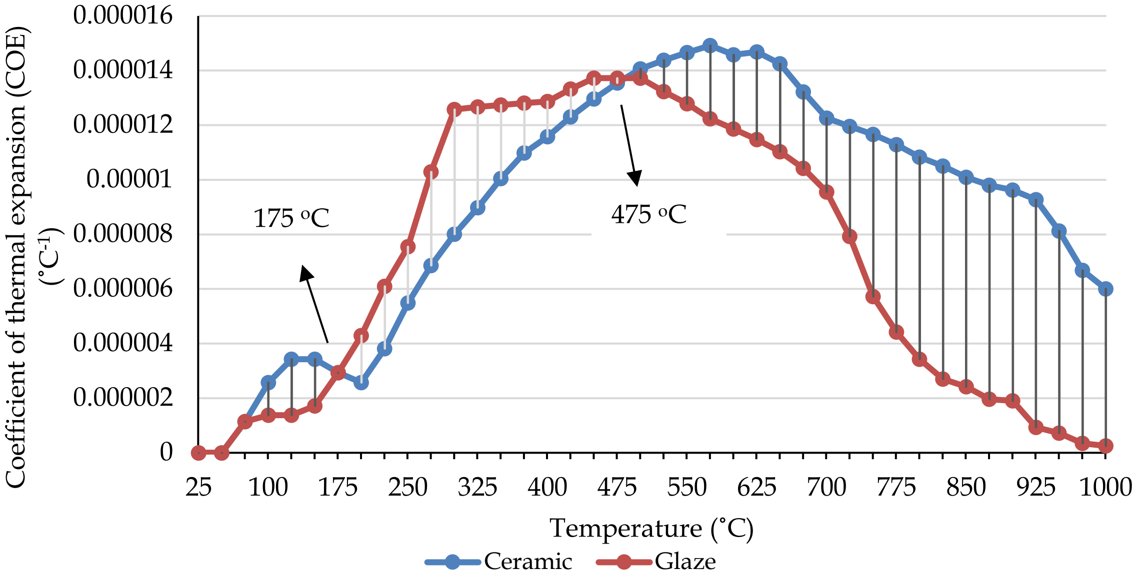

Fig. 6. Coefficient of thermal expansion (COE) sample GB18

The expansion increases of the glaze and coated ceramic samples GB 18 are balanced at 175 °C and 475 °C temperatures. This result means using this basalt glaze for stoneware ceramics is safe at a max temperature of 475 °C without experiencing potential cracks or defects on the glaze surface due to expansion due to high temperatures occurring in glazes and ceramics. At a temperature of 25 °C to 475 °C, the maximum difference in COS value between glaze and ceramics is 1,667%.

3.7. The microhardness of glaze ceramics

The hardness test of the glaze layer was carried out using microvickers, and the average result obtained from 5 indentation points was 6.5 GPa. Several studies divide the hardness type into two: the soft type with a hardness value of 2-3 mohs and the hard type with a hardness value of 5-6 mohs. Hardness values are used for quantitative tests for ceramic products (Cheng et al., 2012). Therefore, comparing research results and other glazed ceramic products is essential. The hardness value of the Ababi basalt glaze layer exceeds the standard hardness value of glaze products commonly available on the market, which is around 1.18 GPa (Gláucia et al., 2021). The hardness results of the ceramic glaze from basalt rock also have a higher hardness value than the porcelain glaze hardness of 2.48 GPa, as made by Cheng and Coworks (2012). According to Mihardi et al. (2012), the dominance of the Gahnite phase formed in the glass-ceramic system affects increasing hardness. High-phase transformation of gahnite must be formed so that the mechanical properties of glass-ceramic are increased (Mirhadi et al., 2012). The increase in silica to 49% and the formation of Gahnite can improve the mechanical properties of the glaze layer to make it more resistant to friction (Abo-Mosallam et al., 2013).

3.8. The physical characteristic of glaze ceramics

Based on porosity and density tests according to ASTM C373-88 (ASTM C373-88, 2006), the porosity value is 0.0%, and the density of the glazed ceramic body is 1.91 g/cm^3, with no change before and after the specific gravity test. Compared to the other paper, the density of glaze made from basalt with the firing method was 1.45-1.5 g/cm^3 according to the used of basalt content 45-77% (Dvorkin and Galuskho, 1969). But in other research, the density of basalt glaze was 1.8 g/cm^3 (Prstić et al., 2007) and

2g/cm^3 (Andrić et al., 2012). The difference in density values is due to differences in composition chemistry, basalt composition, and the process temperature used.

3.9. Surface morphology of glaze ceramics

Based on Fig. 7, the thickness of the glaze formed on the sample has an average thickness of 79.82µm. Judging from the surface morphology, the glaze formed is flat and not wavy. The ability of the glaze formed can also stick perfectly to the ceramic body; this is characterized by the area of interaction between the glaze with the ceramic body being quite extensive, and no cavitation occurs in the interaction area. With a thickness of less than 100 µm, the vitrification process in the glaze layer is well-formed without experiencing cracks in the interaction area or glaze surface (Ozturk et al., 2022; Shi et al., 2019; Wang et al., 2019). The glaze formed has no defects on the surface or in the glaze itself, so it can perfectly coat the entire surface of the ceramic body and is in perfect harmony with the COE results.

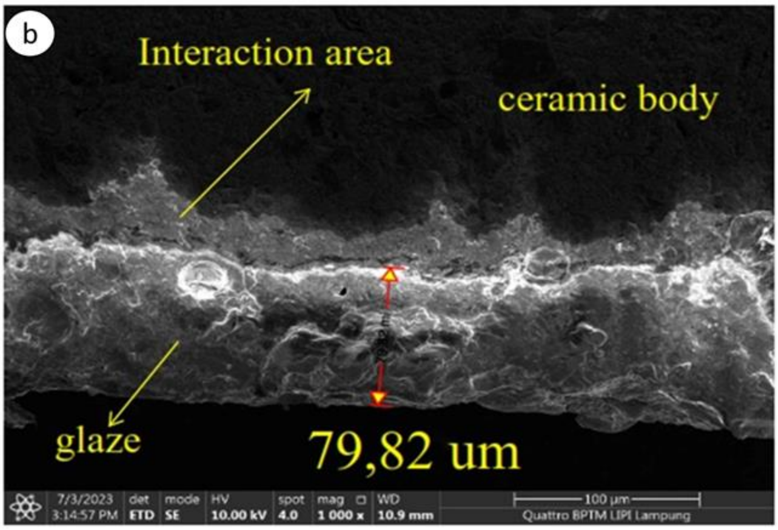

Fig. 7. The morphology of glaze ceramics

4. Conclusions

Basalt rock in the Ababi area, Karangasem Regency, Bali Province, which contains feldspar, quartz, pyroxene, and iron minerals, can be used as raw material for a ceramic glaze with a reasonably high iron oxide (Fe_2O_3) content of around 20.072%. Apart from Karangasem basalt rock as the primary raw material, feldspar, kaolin, and zinc oxide (ZnO) were also used, and three variations of the glaze formula coded GB 8, GB 15, and GB 18 were prepared. From the results of burning the glaze formula on the stoneware ceramic body test object at a temperature of 1,250 °C using a laboratory scale Nabertherm electric furnace, the result of burning glaze code GB 18 was the best formula with a faded yellowish-brown glaze color and a slight growth of crystals on the surface layer of the body being glazed. The glaze needed to be more transparent and more robust (mat). The Vicker's hardness value for the Ababi basalt-based glaze layer in Karangasem Regency is 6.5 GPa, exceeding the standard hardness value for glaze products commonly available on the market, namely around 1.18 GPa. The mineral content in the GB 18 code glaze is gahnite ($ZnAl_2O_4$) and Coesite (SiO_2), which can improve the mechanical properties of the glaze layer, chemical resistance, and friction resistance.

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