EFFECT OF MODIFYING THE HEATING TEMPERATURE ON THE BASALT GLAZE MIXTURES QUALITY FOR STONEWARE CERAMICS

PENGARUH MODIFIKASI SUHU PEMANASAN PADA KUALITAS CAMPURAN GLASIR BASALT UNTUK KERAMIK TEMBIKAR

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ABSTRACT

Research has been conducted on the effect of temperature variations on basalt-based glaze mixtures for stoneware ceramic applications using temperature variations of 1100, 1200, and 1300°C. This research aims to determine the optimum temperature for the best quality basalt glaze. The glaze sample was made using raw materials of basalt, kaolin, and feldspar their composition around 60%, 10%, and 30% wts respectively performing their grain sizes under 100 mesh. Material characterization was carried out by analyzing their XRF, XRD, and optical microscopy. At a burning temperature of 1200°C, the basalt-based glaze mixture significantly influences the structure and changes of glaze on the surface of the specimen from a macro-structural perspective. At the temperature of 1200°C, the glaze layer has reached the perfect melting point and coats the specimen surface evenly and results in not easily cracked and broken. It was proven that the glaze liquid could penetrate the pores, completely covering the surface morphology of the test object. Regarding the multitude of colors formed at temperature of 1200°C, it can optimize the content of dye metals such as iron, manganese, and cobalt in the glaze materials.

Keywords: glaze, basalt, temperature variation, stoneware, coating.

ABSTRAK

Telah dilakukan penelitian efek perubahan suhu pada campuran glasir berbasis basalt untuk aplikasi keramik batu menggunakan variasi suhu 1100, 1200 dan 1300°C. Penelitian ini bertujuan untuk menentukan suhu optimum untuk menghasilkan kualitas terbaik glasir basalt. Percontoh glasir dibuat menggunakan bahan baku basalt, kaolin, dan feldspar dengan komposisi 60%, 10%, dan 30% wt dan ukuran butir di bawah 100 mesh. Karakterisasi material dilakukan dengan menganalisis XRF, XRD, dan pengamatan mikroskop optik. Pada suhu pembakaran 1200°C, campuran glasir berbasis basalt memiliki pengaruh yang signifikan pada struktur dan perubahan glasir pada permukaan percontoh dari perspektif makrostruktur. Pada suhu 1200°C, lapisan glasir telah mencapai titik peleburan sempurna; glasir terbukti mampu meresap ke dalam pori-pori, menutupi morfologi permukaan benda uji dengan baik. Pembakaran pada suhu ini juga mengoptimalkan kandungan logam pewarna seperti besi, tembaga, dan kobalt yang terkandung dalam bahan glasir.

Kata kunci: glasir, basalt, variasi suhu, batu, pelapisan.

INTRODUCTION

Ceramics are widely known throughout the world. In society, the meaning of ceramics tends to be the type of floor tiles. The word ceramic originally came from the Greek word 'keramos' - a form of clay that has undergone burnina process (McColm. а 2013). Dictionaries and encyclopedias defined ceramics as a work of art and technology to produce the objects from fired clay, such as pottery, tiles, porcelain, etc. Ceramics are inorganic solids made from clay and need a combustion process (Rattanavadi, 2018).

Based on material and firing temperature, the ceramics are divided into stoneware, pottery, and porcelain. Stoneware itself is a type of ceramic that is fired at temperatures between 1150°C and 1350°C. This ceramic type is not easily penetrated by water (Rangkuti, Pojoh and Harkantiningsih, 2008). The stoneware is widely used in household and manufacturing ceramics industries. Pottery is made from clay, formed and heated at temperatures ranging from 1000 to 1150°C, so it has a relatively rough and porous surface. Ceramic surface glazing was carried out to beautify the surface appearance and protect and increase the strength of the ceramic body (Lawrence and West, 1982). The last one, porcelain that has translucent characteristicsis made from various mixtures of feldspar, glass minerals, and granite with fine white clay, and is then forged and fired at temperature of 1200 to 1450°C.

Historical and scientific evidence reveals that the earliest glaze, made from wood ash was applied to green stoneware in China (Huang et al., 2020). This proves that the technology for making the glaze has been used since the ancient times. Glaze material consists of three parts, namely silica (SiO₂), flux, and alumina (Al₂O₃). These three components must be balanced with the correct flux ratio to melt silica and alumina for clear glaze (Budiyanto et al., 2008a, 2008b) and require high firing temperatures (Boch and Niepce, 2007). However, the price of the glaze flux materials is guite expensive (Sudarvanto and Agustinus, 2006). The abundant basalt resources in Lampung and its low price make it possible to develop it. Basalt is not only utilized as coating material (Ateş et al., 2017), stone wool fibre (Isnugroho, Hendronursito and Birawidha, 2018), beautiful ornamentation (Hendronursito

et al., 2019), and clinker material mixture (Suharto et al., 2020) but may also be used to produce glass-ceramics with distinct microstructure and various characteristics through phases of thermal processing and chemical composition modification due its mineral content (Candra et al., 2020; Lima, Zorzi and Cruz. 2022). This allows the application of these materials in the most different fields such as coating material (Birawidha et al., 2023). This research investigates the effect of heating temperature modification on basalt glaze mixtures for stoneware ceramics.

METHODOLOGY

The materials used in this study were basalt, kaolin, and feldspar. Raw scoria basalt came from Sukadana, East Lampung, Indonesia. It resembles a sponge with holes around the surface of the rock, has a dark gray appearance, and has a rough texture. With this characteristic, visual identification becomes easier than other basalt rock types. The kaolin and feldspar materials were supplied from the local mines. Kaolin is white and has a delicate texture, while feldspar is blackish-gray in colour and gritty texture.

In this investigation, the preparation of raw materials was begun with destruction of basalt using a Molino de Bolas ball mill for 4 hours. Then a sieving process was carried out to get the powder passing through 100 mesh using the Endecotts brand sieve shaker. The glaze sample was made with a composition of 60% wt of basalt, 10% wt of kaolin, and 30% wt of feldspar, considering that basalt with this composition effectively covers all the pores of the ceramic surface (Andrić et al., 2012). The glazed solution was prepared by mixing all raw materials, adding 120 ml of distilled water, and stirring manually for 20 minutes until the solution was homogeneous. A stoneware with dimensions of 21x14x7 cm was immersed in a glaze solution for 5 seconds until the laver stuck perfectly, then baked in the oven (Phillips) at 100°C for 2 hours. The glaze samples were fired at three temperature figures, namely 1100, 1200, and 1300°C, using a Naberthem furnace with an hour holding time. Figure 1 represents the experimental process of stoneware ceramic glaze production.

Characterization Tools

The characterization analysis in this study was XRF, XRD, and optical microscopy analysis. The chemical composition of raw materials and glaze samples was characterized using the XRF (Epsilon 4 XRF Spectrometer - Malvern Panalytical) that was operating at 50 kV and 3 mA. Crystallinity and phase of raw materials and glaze samples were carried out through XRD (PANalytical Xpert 3) with Cu-K as an X-ray source operating at 40 kV and 30 mA. To determine the morphology of the glaze sample, an Olympus CX23 binocular light optical microscope was used under a magnification of 100x.



Figure 1. Experimental Process of Glaze Sample Production

RESULTS AND DISCUSSION

Results of XRF characterization of the glazed materials are shown in Table 1. The main

components of basalt are SiO₂ at 44.830% wt, Fe₂O₃ at 19.462% wt, and Al₂O₃ at 15.966% wt. SiO₂ comes from raw basalt at 45–52% wt (Singha, 2012), which functions glazing agent in glazes. Fe₂O₃ affects the glaze's appearance, and Al₂O₃ impacts the melting ability or maturity of the glaze product.

The table indicates the main composition of feldspar is 55.859% wt of SiO. Feldspar also contains metal elements such as Fe₂O₃ as much as 7.966% wt. Its functions as a coloring element: other elements include Al₂O₃ (23.885% wt.), and K_2O (9.541% wt., which functions as a melting agent (Supriyadi, Cingah and Suardana, 2012). The feldspar contains silica and has a flux of alumina content, which is suitable as a glaze material at high temperatures (Kronberg and Hupa, 2019). The main composition of kaolin is also SiO₂ 70.247% wt and Al₂O₃ 14.935% wt. These three raw materials are the essential components of glaze and meet the requirements for glaze-making ingredients such as silica, alumina, and flux. The XRD tests of raw basalt, as seen in Figure 2, shows that the raw basalt has crystalline characteristics and a pyroxene phase (Ca-Mg-Fe-Na-Al-Ti Silicate, cod data = 01-086-0162). olivine (Mg-Fe Silicate, cod data = 01-083-1485), albite (Na-Al Silicate, cod data = 01-070-3753), and anorthite (Ca-Al Silicate, cod data = 01-089-1462).

According to Chen *et al.* (2017), most of the crystalline phases that make up the basalt rocks are plagioclase, pyroxene, and olivine. They were complex, brittle, and had an aphanitic structure. Diffractogram of the kaolin (Figure 3) shows the microcline phase (P-Al-Silicate, cod 01-076-0918 and anorthite phase (Ca-Al Silicate, cod 01-085-1660). Kaolin comprises kaolinite in anorthoclase group forms and a small amount of feldspar microclines (Mitrović and Zdujić, 2014). The crystal structure known as microcline also serves as a glaze thickener (Effendi, 2004).

Table 1. Chemical compositions of basalt-based glazes

Chemical Composition (% wt)	SiO ₂	Fe ₂ O ₃	Al ₂ O ₃	CaO	K₂O	TiO ₂	P_2O_5	MgO	MnO	ZrO ₂	Eu ₂ O ₃	LOI
Basalt	44.830	19.462	15.966	13.011	0.691	1.862	0.711	2.487	0.448	-	0.119	0.413
Kaolin	70.247	4.004	14.935	1.990	6.955	0.416	0.864	-	0230	-	-	0.359
Feldspar	55.859	7.966	23.885	-	9.541	1.122	0.854	-	-	0.396	-	0.377



Figure 2. Diffractogram of the basalt



Figure 3. Diffractogram of the kaolin

The feldspar diffractogram (Figure 4) shows its crystalline characteristics, and the phases were microcline (P-Al Silicate, cod 01-087-1789), K-feldspar (K-Al Silicate, cod 01-089-8573), sanidine (K-Al-Silicate, cod 01-080-2108), and cristobalite (Silicon Oxide, cod 01-082-1410). These were usually found in feldspar with a predominant microcline content, often found in feldspar minerals (Haldar and Tišljar, 2014). K-feldspar serves as a smelting agent, but this material is less than optimal in its effect on the combustion temperature, which requires quite a lot of material to lower the temperature (Kronberg and Hupa, 2019; Huang *et al.*, 2021).

Based on the XRF analysis of the glaze samples (Table 2) with 60% wt. basalt, 10% wt. kaolin, and 30% wt. feldspar was dominated by SiO_2 at 45.589% wt., followed by Fe_2O_3 at 18.874% wt., and Al_2O_3 at 16.530% wt. Four primary oxides of the glaze

materials, namely SiO₂, Fe₂O₃, Al₂O₃, and CaO accounted for 91.4% of the total weight, while a lesser proportion of other oxides were present.

Figure 5 shows the dominant structure of the glaze fired at 1100°C is amorphous. Some crystal structures, such as the augite phase (Ca-Mg-Al-Silicate, cod 01-078-1391 and pyroxmangite (Mn-Silicate, cod 01-076-0628), were formed. Both belong to the clinopyroxene group $(Ca,Na)(Mg,Fe_3+AlFe_2+MnTi)(SiAl)_2O_6$

(Zanazzi *et al.*, 2008; Dygert *et al.*, 2014). The crystal form was monoclinic, blackish green or blackish brown, as the main mineral in gabbro, dolerite, and basalt. This mineral was formed in high-phase metamorphic rocks (Zhang et al., 2011). Pyroxmangite does not affect luminescence behavior, resulting in different luminescence faults and variations in light tone (Abo-Naf and Marzouk, 2021).



Figure 4. Diffractogram of the feldspar

K₂O

3.7

TiO₂

1.8

P₂O₅

0.89

MgO

1.08

MnO

0.4

ZrO₂ Eu₂O₃ LOI

0.48

0.17 0.11

CaO

10.4

600-	K4 100	1100		٩	<u> </u>	A
	A= Augite Py= Pyroxm	angite			4	88
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Chemical composition of glaze material Table 2. SiO₂

45.6

Fe₂O₃

18.9

Al₂O₃

16.5

Chemical Oxide

Composition (% wt)

Figure 5. The diffractogram of glaze at 1100°C

Figure 6 shows the diffractogram of a glaze sample fired at 1200°C. The glaze structure amorphous was mostly with minor crystallization. There was a coesite phase (Silicon Oxide, cod 01-083-1833) and lime (Ca-Oxide, cod 00-004-0777). Coesite belongs to silicate minerals with a gray appearance, has a monoclinic crystal structure with a Mohs hardness scale of 7.5, and has a brittle texture with a glass-like sheen (Abdi, Ghalandarzadeh and Shafiei Chafi, 2021). While lime has a white appearance, it has a monoclinic crystal structure and a brittle texture with a glass-like sheen (Kusmiyarti, 2016).



Figure 6. The diffractogram of glaze at 1200°C

Glazes diffractogram with 1300°C combustion (Figure 7) shows amorphous characteristics and several crystal structures. Those were hercynite (Fe-Mg-Al-Oxide. cod 01-089-6898), and quartz (Silicon Oxide, cod 1-078-1257). Hercynite is a group of spinel minerals with dark blue, green, yellow, and brown characteristics. Its texture was like glossy glass to dull with an isometric crystal system (Barbosa et al., 2006). Quartz, known as a member of the silicate mineral group, has a white appearance and a hexagonal crystal structure made of crystallized trigonal silica. The morphology analysis of the glaze samples is shown in Figures 7, 8, and 9.



Figure 7. The diffractogram of glaze 1300°C

As seen in Figure 8, after being fired at 1100°C, the glaze coating looks imperfect. The macro appearance was obtained using an optical microscope with a magnification of 100X (8b) and exhibited a brick-red glaze surface color. The sample surface looks rough; there are still raw glaze grains, and the color combination tends to be uneven (8a); the immature surface related to the Al₂O₃ component in the glaze hinders crystallization due to its more significant influence on viscosity (Pekkan, 2015). The dotted pattern that appears on the surface looks like the effect of the Fe, and Mn content (RoquéRosell *et al.*, 2021). Figure 8(c) shows the thickness of the glaze sample during a 5-second immersion is about 223 μ m. The glaze layer did not blend very well with the stoneware body and precisely cover the pores.

After being fired at 1200°C (Figure 9), the glaze laver forms look perfect and dark matte in color. Figure 9(a) shows that the surface is even, coats the test object's surface uniformly, and has a smooth appearance. The immiscibility of stable fluids in multicomponent Fe-rich basaltic-andesite compositions occurs at temperatures up to 1200°C (Hou and Veksler, 2015). The sample surface under 100 Хmicroscope magnification was a dominant brown-redblack color, as shown in Figure 9(b). As seen, the attractive yellow hue is evenly distributed between the brown-red dominance, which is usually caused by Fe, Ti, and Zr content (Molinari et al., 2020; Figure 9c). The white droplets contain a lot of calcium, in comparison to the bigger spherical droplets in calcium-rich areas of the glazing, increased CaO causes a more significant droplet volume percentage and higher P2O5 (Yuan et *al.*, 2022) The glaze thickness was about 416 μ m after 5 seconds of immersion; the glaze layer filled the pores and completely covered the stoneware body.

After being fired at 1300°C, the pores of the stoneware were evenly coated and wholly covered with the basalt glaze. Firing glaze at 1300°C reduces pin-holing defects on the glaze surface because the feldspar content in the glaze material as flux oxide will reduce the viscosity of the glaze, making it easier for gas components to escape from the glaze surface (Hasanuzzaman, Islam and Rashid, 2022). It can be seen that the blackish brown was dominant with some yellowish colors. Under 100X magnification (Figure 10), the iron element predominates with a red-black color with a slight yellow tint from low TiO₂ (Wood, 2021). The glaze thickness was 203 µm, obtained during immersion for 5 seconds, similar to the thickness of the glaze which fired 1300°Č 1100°C. At combustion at temperature, the ceramic body's densification is also accomplished (Hasanuzzaman, Islam and Rashid, 2022).



Figure 8. Morphology of 1100°C glaze samples at 100X magnificent: (a) after heating, (b) glaze structure (c) glaze thickness



Figure 9. Morphology of 1200°C glaze samples at 100X magnificent: (a) after heating, (b) glaze structure (c) glaze thickness



Figure 10. Morphology of 1300°C glaze samples at 100X magnificent: (a) after heating, (b) glaze structure, (c) glaze thickness

CONCLUSION AND SUGGESTION

The effect of a firing temperature of 1100°C on the basalt glaze produces an immature glaze layer. Firing basalt glaze at 1200°C produces a mature layer and a variety of color shades; the temperature allows the basalt glaze to fill the pores and cover the surface of the stoneware. The thickest layer of glaze obtained at this temperature was 416 µm. Meanwhile, firing the basalt glaze at a temperature of 1300°C makes the glaze layer seep evenly into the pores of the ceramic because the feldspar content reduces the viscosity of the glaze solution. This temperature reduces the basalt glaze's needle-hole defect and coats the stoneware's surface smoothly until the layer is firmly bonded.

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