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Case Study of Ceramic Firing Profile for Terracotta-Based Glaze

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Abstract

Terracotta clay is a natural resource that has the potential as a raw material in the ceramic field. This study investigates the effects of color glaze using terracotta clay powder as an alternative substance for ceramic colorants in glaze formulation. Terracotta clay powder is proportionately added to glaze formulations to determine the potential for color added to the glaze at temperatures ranging from 1140 °C to 1200 °C. The content in terracotta clay is bounded with potash feldspar, silica, zinc oxide, and calcium carbonate. Results show the apparent color of the surface on the ceramic glaze samples with different effects.

Keywords: Terracota; firing profile; glaze

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1.0 Introduction

In ceramics, exploring material characteristics and the capability to create a construction form is normally practiced by artists or creative thinkers (Anwar, 2016). Especially during the crafting process of a design form (Awang et al., 2016; Anwar et al., 2015; Vermol et al, 2011). In this research, there is a potential gap to be fulfilled in promoting one type of clay-based material to be discovered which is known as terracotta. Terracotta clay is also called “red clay”. This is because when the clay is fired, it becomes reddish brown, also known as the terracotta color. Terracotta clay contains around 9 percent iron oxide which creates the typical red-brown fired color (Fraser, 1998) and it is a naturally-formed clay. Clay minerals are considered popular minerals in the production sector because they form the basis and building bricks of pottery. The clay properties are specified by the fact that they are layer materials. In ceramics, minerals are not limited to certain ceramics produced. Clay minerals, for instance, can have many by-products such as clay bodies, slip clay, and surface decorations. It can also produce natural colors for ceramic products. Yahya et al. (2013) stated that the color of the ceramic body depends on the iron amount in the mixture and also the way the iron is combined with clay minerals and additives. It also depends on what compounds are formed when we fire the ceramic body. Clay minerals need to be bound with other materials as they cannot rely only on a single base (Fournier, 2000).

The use of natural resources such as terracotta soils can also be used as dyes in ceramic products. With this, color can be diversified. The main purpose of this research is to determine the potential of the composition of the main material for terracotta soil to be used as pigments in ceramic glaze. The method to be used in this research is experimenting with the terracotta soil itself to be used as a pigment, such as testing the composition of the terracotta soil, creating test pieces, temperature firing, and the maturity of glazes according to their

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suitability. It can also be used as a decoration material on ceramic products. The surface of the ceramic body coated by a layer of glaze is smooth and shiny. In addition, colorant pigment is also added to considerably enhance the glaze on the ceramic body. In addition, glazes also have optical properties such as translucent, transparent, and opaque. Glazes can also be used as protective coating, markers, and coatings for sanitary products, culinary, tiles, industrial tools, and household items.

The choice for Sayong terracotta clay was made because the natural resources in Sayong, Perak, are very easy to obtain and can be acquired in large amounts. It is also cheap, and the quality is up to standards for ceramic and pottery making. Looking at the potential of terracotta clay which has been used for generations, it has the potential to be developed and expanded in usage and functionality, such as ceramic colorants and glazes, as well as its decoration techniques. Terracotta is chosen due to the potential of its contaminant property, as it acts as a flux, making the material fire much harder than a mix of white-burning clays at the same temperature would. Engineers refer to the degree to which clay has been fired as its "maturity" (Hansen T., 2017). Iron oxides dye the ceramic body red. According to Kreimeyer (1987) and Anwar et al. (2011), red tones become weaker when more iron is incorporated into the structure of the compounds and less remains in the iron oxides. Mention by (Jordanova, 2019) Iron oxides, especially magnetite (Fe_3O_4) and hematite (α-Fe_2O_3) are the key minerals for tracking the thermal transformations undergone by burnt clay materials. As with this study, the first step is to test the raw materials to identify their mineral composition and conduct research on the suitability of mixing terracotta with glaze materials (simple additions to base glaze), including a view on fluxes and the percentage of terracotta to mix with colors for the glaze. Mineralization, according to Robert Fournier (2000) and Awang et al. (2016) explained minerals as the earth's cooling and subsequent weathering, breaking down, and recombining. The combinations of elements have distinctive physical and chemical properties and, in the present environment, a degree of solidity.

2.0 Method

2.1 Porcelain Test Pieces Preparation

Large chunks of raw porcelain material were initially crushed to smaller sizes with a mallet (between 1.0 and 2.0 cm). The container was then filled with 23 kg of crushed porcelain clay and 14.5 liters of water using a handheld mixer. The mixing technique resulted in a consistent slip. After 24 hours, the soaking process was repeated.

After being soaked in a porcelain slip and transformed into semi-dry, leather-hard clay, it is placed on the plaster of Paris to dry. This technique took 5 hours to become leather-hard at room temperature. The weather has a big impact on the leather-hard process. The spiral technique is then used to knead the porcelain clay process to remove any air bubbles.

The test pieces measure 2 inches broad by 2 inches high. The test pieces are small and flat measuring half an inch in shape. The sloping part's form is moderately wide, measuring 1 inch. Figure 1 depicts a selection of test components. The test pieces have a flat and a concave section to assess the fluidity (running) of the glaze on the test pieces. The gradient on the shape of the sample is used to observe the fluidity (flow) of gel on the sample's surface. After the test pieces' shapes have dried, the press mold is created using plaster of Paris (POP). POP for the press mold is made up of 200 grams of plaster of Paris and 250 milliliters of water. The thickness of the plaster mix can result in a high-quality mold.

Before the press mold technique, the mold must be cleaned to remove any debris and dust from the surface. Because the surface of the sample will be uneven when using a press mold to make porcelain test pieces, the skills involved in making samples must be rigorous. Technically, this procedure necessitates skill and soil wetness. Before proceeding, previously made test pieces are dried. The same process was used to make two stoneware soil sample test pieces as detailed above.

Fig. 1: the sample of test pieces

2.2 Shrinkage Test

The shrinkage test for porcelain and stoneware clay used in this study was performed to identify the three important general characteristics to take note of shrinkage, absorption, and warping or slumping Yakub et al., (2016). Other important qualities to note are color and texture.

The shrinkage testing was performed using a slab with bar form and having two marking point to measure the shrinkage changes of porcelain clay as shown in Fig. 2.

Fig. 2: The process of shrinkage test using a test bar bar slab porcelain clay

Following the process, the porcelain clay test bar in wet condition is measured for length using a ruler before drying and firing. Furthermore, the porcelain shrinkage test has two stages: shrinkage after drying and shrinkage after bisque firing. The first measurement is taken after the test bar is dry, and the second is taken after the bisque firing process at 900°C. The test bar sample of porcelain clay was measured with a guide point that was marked on the porcelain test bar surface that was made from slab bar to determine the differences in shrinkage between wet and after firing.

The shrinkage percentage of the substrate body can be found in equation 1 with Lb = length of wet condition and La = length after a dry while in equation 2 for shrinkage percentage after firing with formula Lb = length of wet condition and La = length after bisque firing. Compaction is the artificial and mechanical process of rapidly decreasing the volume of soil through the removal of air vacuums, increasing density. Densification of soil happens naturally as a result of foundation soil consolidation caused by pore water outflow caused by structural loads. During the firing process, the minerals contained in clays are structurally and chemically modified that significantly transforming the original clay materials. The results obtained resulted in the substrate body which shrank up to 4.76. % from its original size which is 10.5 cm in length and 10.0 cm after drying while the percentage of shrinkage after bisque firing is 9.52. % from the original size which is 10.5 cm in length after firing (Anwar et al. (2011).

$$\text{Drying shrinkage } (\%) = \frac{(L_b\text{-}L_a)}{L_b} X\ 100\% \tag{1}$$

$$\text{Firing shrinkage } (\%) = \frac{(L_b\text{-}L_a)}{L_b} X\ 100\% \tag{2}$$

Following that, the research proceeded with the creation of a transparent glaze using local terracotta clay powder and one firing profile at 1200 °C for gloss temperature soaking for 60 minutes. The temperature was reduced to the cooling point and then gradually reduced for the cooling procedure depicted in Fig. 4. Table 1 shows how to prepare the colorant glaze formulation's % weight formula. The goal of this research is to look into the possibility of incorporating local terracotta clay into a transparent glaze recipe. To replace the glaze's traditional colorant, the tested local terracotta clay was applied in proportions ranging from 0% to 50%. The weight percentage is chosen based on the reference research. Because it relates to the examination of agents' colorants in a transparent glaze for promoting the color, local terracotta clay powder was employed in the formulation. Furthermore, local terracotta clay is mixed with a different percent of transparent glaze to form the color. Besides that, the percentage additions were used to produce color especially for the temperature glaze firing, with appropriate and accurate calculations in the glaze formulation.

Table 1. Basic formulation of transparent glaze

Raw material (wt%)			
Potash feldspar	Flint	Calcium carbonate	Zinc oxide
33	32	20	15

2.3 Drying and Bisque Firing Process

Test pieces were dried at room temperature for 3 days to bone dry before firing. As shown in Figure 3, the bisque firing was performed at 900°C for four hours with soaking time for 30 minutes to obtain the bisque product for the glaze coating process using an electric furnace (MV Muffle Furnace 1400 type) in an oxidizing atmosphere. The temperature is used to obtain the body of the porous bisque body for the easier glaze to absorb during the glaze application process but with high strength so that it does not fragile easily and break.

Fig. 3: Bisque body was fired in MV Muffle Furnace 1400

Before firing, porcelain test pieces were allowed to dry out completely at room temperature for 4 days. To produce the bisque product for the glaze coating process utilizing an electric furnace (MV Muffle Furnace 1400 type), the bisque firing was carried out at 900°C for four hours with a soaking time of 30 minutes. The temperature as shows in Fig. 4 is used to create a porous bisque body with high strength so that it won't break easily and is easier for the glaze to absorb during the application process.

Fig. 4: Firing profile at 1200oC for gloss temperature.

Local terracotta clay is mixed with a difference of percent with transparent glaze to form the color. Besides that, the percentage additions used to seed color growth, especially for the temperature glaze firing, with appropriate and accurate calculations in the glaze formulation. Furthermore, the percentage for local terracotta clay starts at 20%, 30%, 40%, and 50% added to produce color, whereas impurities added to produce color can occur with a more blended pigments that give unpredictable results after firing.

2.4 Flowing Test of Glaze

The slip-casting method was applied to proceed with the flow testing block. The mold which made from Plaster of Paris (POP). It was purposely prepared as the test block as shown in Figure 5, The block was designed intended to determine the flow of glaze before and after the addition of terracotta into the glaze formulation. The testing block form was performed with two curve holes to indicate the differences in the result after firing.

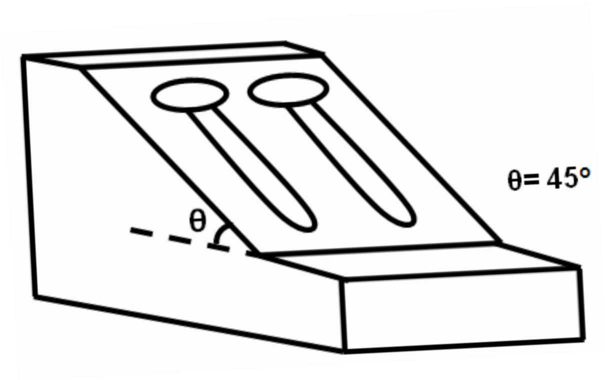


Fig. 5: Flowing testing block

3.0 Results and Discussion

Based on the transparent formulation specified in Table 1, it has four samples for the terracotta glaze recipe (A1–A4). All materials used are the same percentage, except conventional local terracotta clay was added in the range of 20% to 50%. Potash feldspar 33%, silica 32%, zinc oxide 15%, calcium carbonate 20%, and terracotta as a colorant Its purpose is to compare the effects of varying the percentage of terracotta in the transparent glaze. The raw materials were weighed by weight percentage using electronic balancing to obtain a 100g dry weight of the powder, which was added with water (120 ml) and milled in a ball mill machine for grinding and homogeneous mixing. The grinding process takes one hour, and then it is left for 24 hours for the aging process. The bisque product is cleaned first with a damp sponge to remove dust, and the dipping technique is used for the application process.

Table 2 Glazing 1200°C Temperature Transparent Glaze on Porcelain sample

	Sample Code			
Temperature 1200°C				
	A1	A2	A3	A4
Colourant %	20%	30%	40%	50%

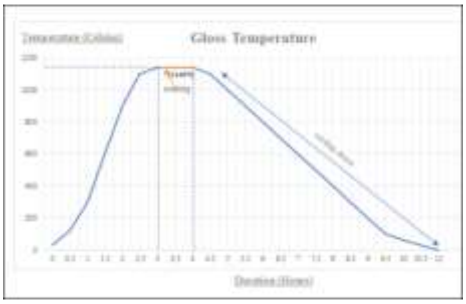
The raw materials were weighed by weight percentage using electronic balancing to obtain a 100g dry weight of the powder, which was added with water (120ml) and milled in a ball mill machine for grinding and homogeneous mixing. The grinding process takes one hour, and then it is left for 24 hours for the ageing process. The bisque product is cleaned first with a damp sponge to remove dust, and the dipping technique is used for the application process.

3.1 The Investigation of Various Gloss Temperature

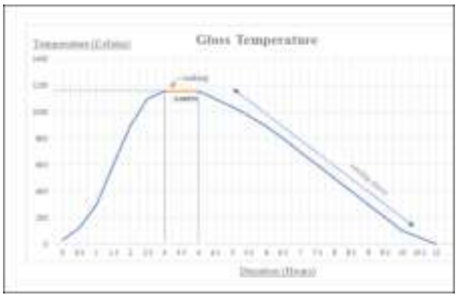
This step was performed to investigate the optimum gloss temperature for this set of glaze formulations (A1 – A4). After completion of the transparent glaze coating process using a dipping technique, the coated surface must be left for drying before the firing process. The early experiment was started with an investigation of gloss temperature and one range of glaze temperature has been chosen at 1200°C with normal cooling and one hour soaking period.

Indicate the firing profile and the soaking period used during the firing process. four range of firing profiles with gloss temperatures (Tgloss) ranging from, 1140°C, 1160°C 1180°C, and 1200°C and respectively duration for each firing profile as shown in Fig. 6. In addition, after the optimum gloss temperatures (Tgloss) were identified, this study continued to investigate the diversity of glaze temperature for identified the differences in effects between all temperatures. For this test the sample code for porcelain (PI, P2, P3, P4) and code samples for stoneware (S1, S2,S3, S4).

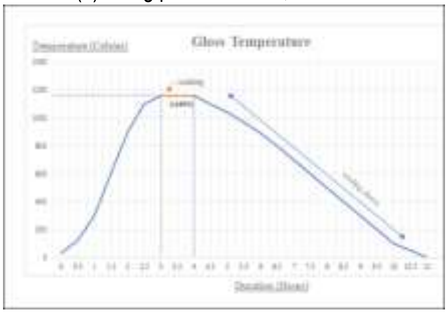
To expand the study about firing temperature, the gloss temperature in the range 1140°C, 1160°C, 1180°C, and 1200°C was performed to obtain the optimum gloss temperature. To begin with, the firing profile for 1140°C (Fig. 6a). It takes one hour and reaches a melting point of 1100°C for gloss temperature with a soaking time of 60 minutes. From gloss temperature, it dropped to glaze temperature at 800°C in one hour and soaked until the furnace shut down for cooling naturally.



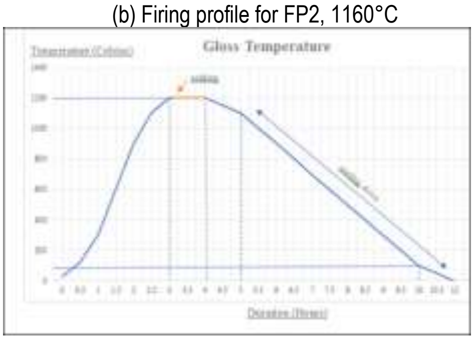
(a) Firing profile for FP1, 1140°C



(b) Firing profile for FP2, 1160°C



(c) firing profile for FP3, 1180°C



(d) Firing profile for FP4,1200°C

Fig. 6: Firing profile for the experimental glaze test

Next, Figure 6 (b) indicates the firing profile for 1160 °C. The gloss temperature takes one hour and 60 minutes, with soaking time at 1160 °C. While the time between the gloss temperature and the glaze temperature decreased, it was soaked for one hour before stopping the firing to allow the temperature to cool. Furthermore, the time required to reach the gloss temperature of 1180 °C is one hour and 60 minutes, as shown in Figure 6 (c). As shown in Figure 6 (d), after soaking for 60 minutes, it takes one hour and 30 minutes to reach the melting point for gloss temperature at 1200 °C. Similarly, after soaking for one hour, the temperature increased from 1140°C to 1200°C, but it did not drop from gloss temperature to glaze temperature until the furnace was turned off and allowed to cool naturally. The furnace used is an oxidizing MV Muffle Furnace 1400-type electric firing furnace. Numerous investigations have been conducted to ascertain how the firing temperature affects the characteristics of ceramic materials. It is well known that the firing temperature has an impact on the flexural strength of ceramic materials.

3.2 Colour Glaze at Various Temperature

The best gloss firing temperature was chosen to be 1180°C, and the optimum glaze formulation samples were chosen to be the P3 formula. The temperature of 1180°C is optimal for gloss temperature since the results demonstrate that the glaze was sufficiently melting and stable. While P3 was chosen as the glaze formulation because the results show that using terracotta powder in the glaze can replace traditional color and produce formation on the glaze surface while also lowering material and firing costs.

4.0 Results

The glaze-flowing test is performed using a flowing test block. Preparation of the samples was done by drying glaze P3 and S3 to obtain the powder form. The glaze was refined using a mortar and mixed with 1 ml water to convert it to a small ball shape. As shown in Figure 7, the powder ball (0.30 g) was placed into a curve shape on the flow test block before the firing process was carried out. The sample will be melting through the drain on the testing block with a 45º degree angle to facilitate the flow of samples when firing process is performed.

Fig. 7: The powder ball was placed into a curves shape on flow test block

The firing process at 1180°C for gloss temperature with normal cooling temperature. MV Muffle Furnace 1400-type electric firing in an oxidation atmosphere was used during the flowing test to determine the difference in the melted glaze after firing. Comparison is made between the Porcelain bodies in glaze formulation. The distance flow of the glaze was measured from the curved hole until the melted glaze ends. This testing is to identify the differences in flowing between P3 and S3 samples and whether terracotta powder has potential or stability for testing the flowing of glaze as revealed before this glaze is extremely flowing during firing. Hence, the sample that shows the most distances flowing in the melt is considered to have the highest flow during the firing process.

The study on terracotta clay powder glaze test concludes with a flowing test of this glaze. Figure 8 shows the result between glaze formulations of sample P3 with 30% raw material terracotta clay powder in a transparent glaze. The firing was performed with 1180°C gloss temperature and soaking at temperature. In addition, the glaze spherulite occurs on the P3 sample. The result indicates the P3 sample flows a very small amount, the sample showed 0.2 cm of glaze flow from starting point. It is caused by the presence of local terracotta clay powder in the glaze which helps to reduce excessive flow in the glaze. As mentioned before, the Al2O3 was used in a transparent glaze and local terracotta clay powder. Local terracotta clay powder indicates 30% of Al2O3 which is higher than aluminum oxide in conventional silica is ..%. Hence, to control the flow of melted glaze and preventing from running off the ware should have a very small amount of alumina around 1 to 2% in the glaze formulation. In short, the result shown by adding 30% local terracotta clay powder in the P3 sample has the potential to reduce the ceramic glaze from running off and flow melt.

Fig. 8: The results of flowing test between P3 and S3

5.0 Conclusions

After all the experiments toward terracotta clay as a colorant glaze with adding terracotta clay had been done, the glaze was applied to the final product to determine the suitability of glaze formulation by adding terracotta clay powder for functional ceramic products bowl and (SME) product labu belalai Gajah. From the observation made for the market, SME products are generally applied to commercial products. As mentioned before this, the glaze should go through the determined color coating and phases to allow the formation of the glaze by extending the firing temperature. Hence, the substrate that is used for colored glaze must have a characteristic shape that makes it easy for the transparent glaze to be applied to surface coating, easily and smoothly. In this study, the product chosen to apply the glaze is called bowl and SME product, labu belalai gajah. The SME Product, labu belalai is a ready-made casting and firing bisque from Mr.Izuddin Bin Isham from syarikat Izue Craft,B12(A), Inkubator, Kraftangan Malaysia Cawangan Perak, Km40 Jalan Ipoh, 33600 Enggor Perak.

(a) (b)

Figure 9 (a) The close range of SME product, labu belalai gajah, fired at 1180°C of gloss temperature. (b) The close range of bowl surface with texture layer shape of terracotta clay,s ayong glaze formation. The top view of bowl fired at 1180°C of gloss temperature

The firing for the final products of the terracotta-coloured glaze bowl and SME product belalai gajah was done as shown in Fig. 9 (a) and Fig. 9 (b). The bowl used porcelain body clay to achieve the optimum results, and the product, labu belalai gajah, used cast stoneware as its SME product. As mentioned before, the formula of terracotta clay glaze was applied to the bowl and fired at 1180° C of gloss temperature. The melting point for the gloss temperature was at 1180° C was held for 60 minutes for the soaking period, and after that, the temperature dropped with soaking time, and the furnace shutdown to allow it to slowly cool down naturally. The purpose of this study was to look at how the firing temperature affected the phase composition and color characteristics of ceramic materials made from nearby terracotta clay.

The application of glaze on a bowl is not a discovery in glaze formation. Layer glaze on the surface of the bowl as shown in Fig. 9 (a) and Fig. 9 (b). For this, we first determined the evolution of glaze aesthetic characteristics as a function of mineral content, establishing the optimum terracotta clay powder addition that would yield a glaze with acceptable aesthetic characteristics.

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