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Effects of Melting Temperature to the Properties of a Ceramic Glaze

Mohd Al Amin Muhamad Nor[1*], Noor Asliza Ismail Adnen[1], Mohd Aidil Addha Abdullah[1], And Mohd Zaky Noh[2]
[1]Faculty of Science and Marine Environment, Universiti Malaysia Terengganu,
21030 Kuala Nerus, Terengganu, Malaysia
[2]Materials Physics Laboratory, Faculty of Science, Technology and Human Development,
Universiti Tun Hussein Onn Malaysia, 86400 Batu Pahat, Johor, Malaysia
*Corresponding author (e-mail: al_amin@umt.edu.my)

Production of low temperature glazes can benefit manufacturers by reducing production costs as well as improving the aesthetical value of ceramic products. A low temperature glaze was prepared by mixing 1.6 g of carboxymethyl cellulose (CMC), 20% boron oxide, 10% kaolin, 45% sodium feldspar, and 25% sodium silicate. Addition of CMC into the mixture was aimed to strengthen the bonds between particles. The mixture was stirred at 900 rpm ± 100 for 1 hour and ceramic pieces were dipped into the prepared mixtures for 10 seconds, then dried and transferred into a furnace to be melted at different temperatures, which was in the range of 800-1100°C. The selected sample was melted at 900°C for 1 to 4 hours of soaking time. Then, the sample was taken out and observed using Table Top Scanning Electron Microscope (TTSEM) and the hardness of the glaze was tested using Wilson Hardness. The dried glaze powder was characterized using X-Ray Diffraction (XRD). It was found that the best melting temperature and soaking time in producing a perfect glaze with no defect was 900°C with 4 hours of soaking time. Hardness test also showed that the glaze was hardened from 92.5 to 93.7 *hv* as the temperature increased from 900 to 1000°C. XRD results confirmed the presence of boron oxide, kaolinite, feldspar, and sodium silicate.

Key words: Melting temperature; ceramic glaze; melting time; hardness

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Surface properties, usefulness, and appearance of ceramic products can be improved by applying a glaze onto the surface. Glaze is a vitreous or glassy coating that has been formulated to fix on ceramic surfaces, obtained by cooling oxides or minerals applied and melted on the surfaces of objects [1-3]. Glass former, flux, and stabilizer are the main composition required in producing either a matte or glossy glaze.

Glass former is an important component in producing a glaze because it gives a glass feature to the glaze and usually has a high melting temperature. Flux acts as melting agent which is required in bringing down the melting temperature of glass former by promoting the fusion or formation of glassy phase at a lower temperature [4]. Stabilizer is used to ensure that the glaze is stable, fixes on the bisque body with no run-off during melting. A glaze requires a right expansion and coefficient with the ceramics. If a glaze contracts more or less compared to the ceramics, it can cause the glaze to either crack or peel off [5].

Glazes have their certain range of temperatures for them to melt. When glazes are fired at an extremely low temperature, the glazes will not melt properly. Meanwhile, the presence of an unbalanced body fit between a bisque body and a glaze when fired at a too high temperature causes the glaze to runs off from the bisque body. Table 1 shows ranges and classifications of melting temperatures of glazes.

Table 1. Ranges of melting temperature glazes [6]

Range of temperature	Melting Temperature
600°C to 1050°C	Low
1000°C to 1050°C	Medium
1200°C to 1400°C	High

A study in producing a low temperature glaze had been carried out [7]. The finding showed that a glaze from a mixture of Phuket municipal solid waste, soda lime cullet, and borax was successfully produced at a low melting temperature, which was at 950°C. The composition percentage used in this study was inspired by the composition used by Makishima and Nagata [8]. They found that the system of Na_2O-K_2O-CaO-ZnO-$A1_2O_3$-SiO_2 was able to lower temperature by 50 to 200% depending on the amount of metal oxides used for replacement.

Development of glaze coating on the surface of ceramic bodies has gained an interest among ceramic manufacturers due to its low cost, higher hardness, and an improved mechanical property [9,10]. Low temperature glazes can give benefits to manufacturers by reducing production costs, as well as energy saving. Addition of suitable amounts of flux into the system can lower the melting point of glazes, resulting in the formation of low temperature glazes.

Numerous studies on producing low temperature glazes had been done [7,8]. In an effort to further explore the possibilities offered by this composition, the effects of melting temperature towards the surface morphology of glaze entity forms and different soaking time as well as hardness of melted glazes were carried out in this study.

EXPERIMENTAL SECTION

The glaze formulation was prepared using 20 wt.% boron oxide (Sigma Aldrich), 10 wt.% kaolin a (Kulim High Tech Sdn. Bhd.), 45 wt.% feldspar (Kulim High Tech Sdn. Bhd.), 25 wt.% sodium silicate, and 1.6 g of CMC (Fisher Scientific). The formulated glaze was added into a 250 mL beaker containing 60 mL of distilled water and stirred using a mechanical stirrer (KIKA LABORTECHNIK, RW 20.n) for 1 hour, forming a well-mixed glaze mixture. Ceramic pieces (6 cm × 2 cm) were dipped into the glaze mixture for 10 seconds before being dried in an oven overnight at 60°C and transferred to the furnace. The samples were melted at different temperatures that ranged from 800 to 1100°C for 2 hours with the heating rate of 5°C/min.

Dried glaze powders were characterized using XRD (Rigaku Miniflex (II)). The morphologies of the glaze samples were observed using TTSEM (Hitachi, TM1000) and the hardness of the samples was measured using Wilson Hardness test.

RESULTS AND DISCUSSION

The study on the effects of melting temperature was carried out at temperatures of 800, 900, 1000, and 1100°C, with the heating rate of 5°C /min and 2 hours of soaking time.

X-ray Diffraction Analysis

In Figure 1, X-Ray diffraction patterns with identified elements of the analyzed glaze powder are shown. The mixture sample contained 20% boron oxide, 10% kaolinite, 45 wt.% feldspar and 25 wt.% sodium silicate.

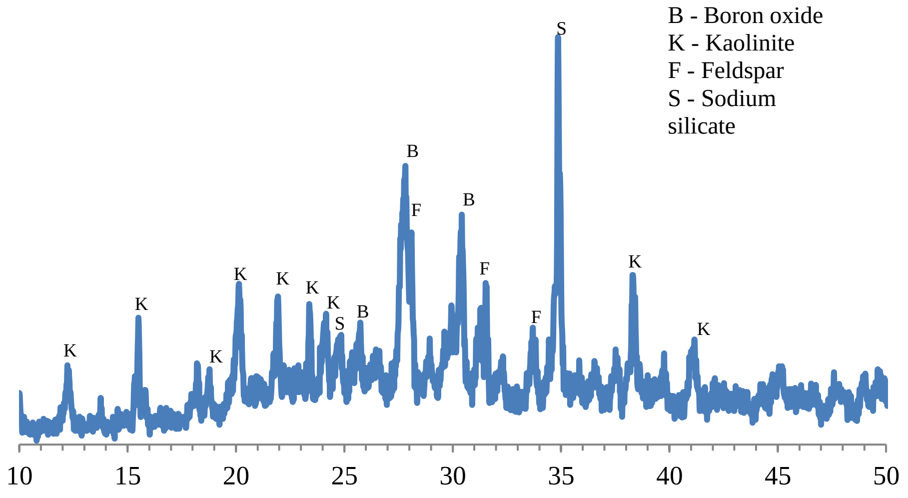

Figure 1. XRD patterns of the formulated glaze

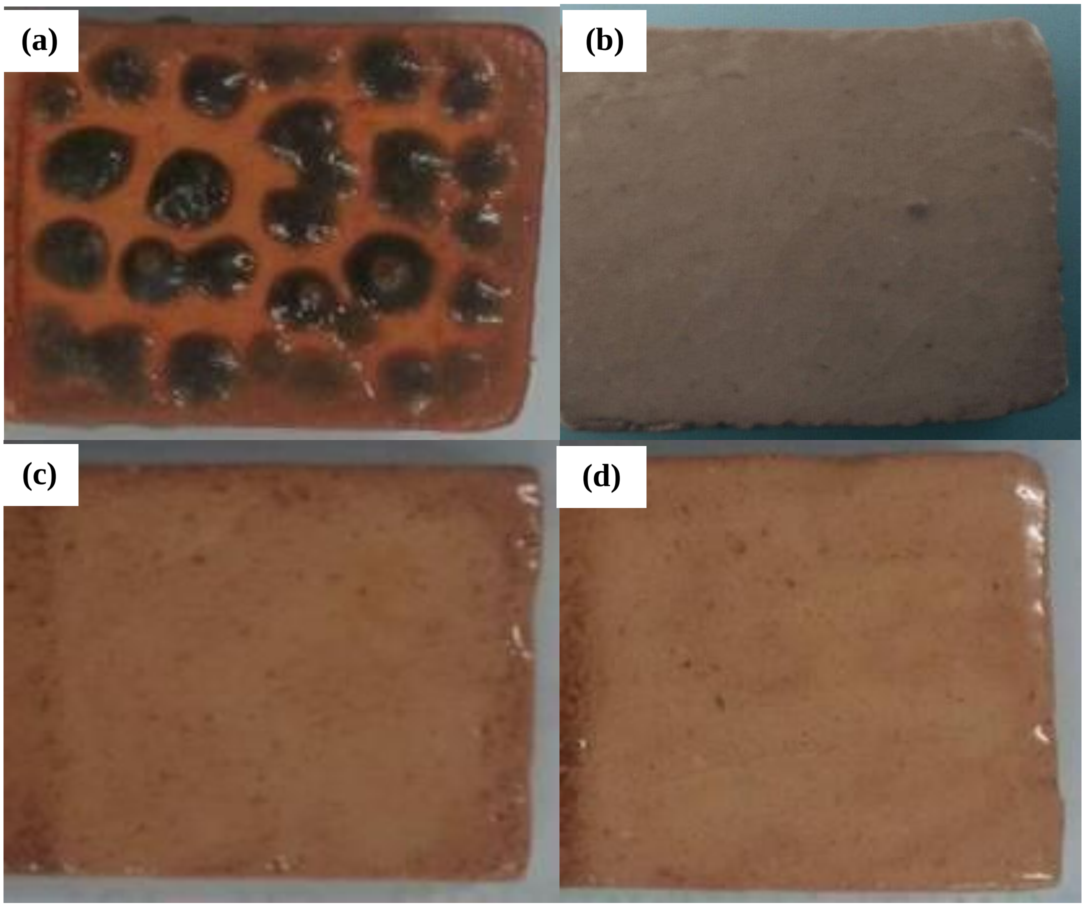

Figure 2. Glazes melted at different temperatures; (a) 800°C, (b) 900°C, (c) 1000°C, and (d) 1100°C

Physical Observations on Glaze Surfaces

The fluidity of a glaze is related to the defects that occur after firing such as pin holing, crawling, crazing, blistering, and leaching. Figure 2(a) illustrates that the glaze did not properly melt at 800°C, which left defects such as pin holing. This phenomenon occurs when the firing temperature is too low and gives the glaze insufficient time to melt completely.

As the temperature increased to 900°C, the glaze was glossy and melted completely. However, defects such as crazing appeared on the surface of the glaze, may be due to thermal expansion mismatch. It showed a network of lines on the surface of the glaze. Crazing is considered a glaze defect due to presence of vessels in glaze that may weaken the glaze and also become a breeding ground for bacteria or gem [11] and it happens due to different thermal expansion between the glaze and body [2]. Compatibility between the ceramic body and glaze is extremely important as all fired ceramic products will experience expansion and contraction during heating and cooling.

The temperature was further increased to 1000 and 1100°C; it was observed that the glaze melted completely giving glossier surfaces with no defect. It could be concluded that 900°C emerged as the best temperature where the glaze starts to completely melt as shown in Figure 2.

Hardness Test

Hardness of ceramic glazes can be affected by porosity, microstructure, and also composition of residual glassy phase [12]. Figure 3 illustrates the hardness values for the glaze melted at different temperatures. It was found that the hardness of the glaze slowly increased from 92.5 to 93.7 *hv* as the sintering temperature increased from 900 to 1000°C. It could be concluded that a glaze without crack possesses higher hardness compared to a cracked glaze. The thermal expansion coefficient mismatch caused crack and affected the surface hardness of the glaze layer [13]. As the temperature increased from 1000 to 1100°C, the glaze became completely homogeneous and this hardened the glaze as cooling process took place. It is in agreement with [14], in which the hardness of glazed ceramics increased with the increase of sintering temperature. Other than thermal expansion, parameters such as porosity, amount and type of crystalline phases, microstructure as well as composition of residual glassy phase can also affect the micro-hardness of glass-ceramic glazes [13]. However, the result for the glaze melted at 800°C was not collected because the glaze was not tested due to its unmelted properties.

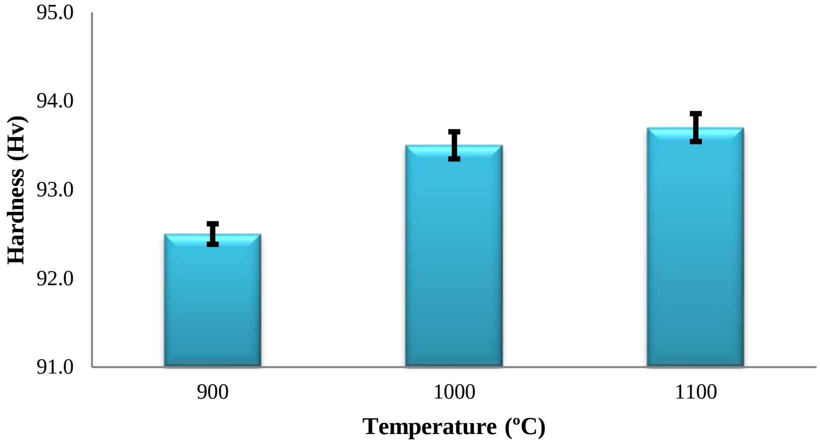

Figure 3. Hardness of glazes using hardness glaze

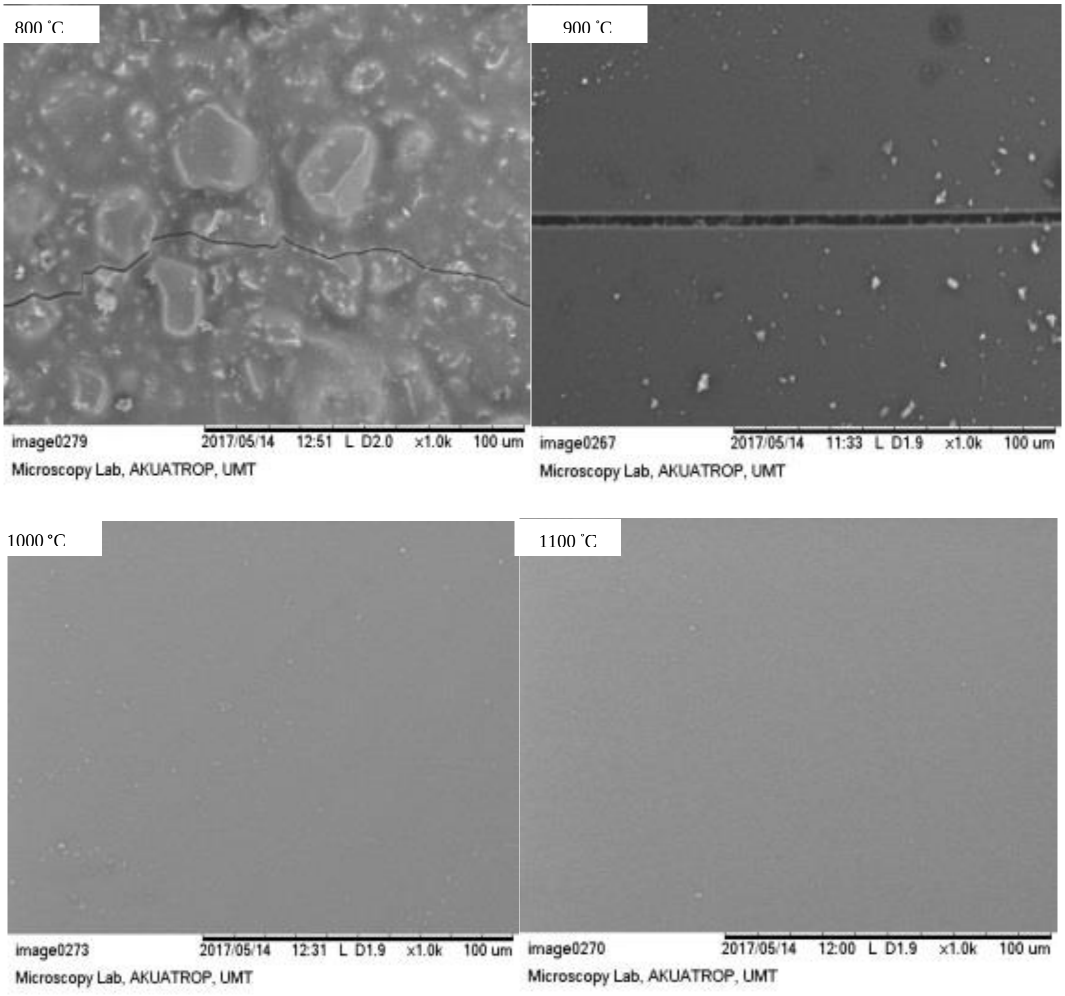

Figure 4. TTSEM micrographs of glazes melted at different temperatures

Morphology of Glazes

Effects of different melting temperatures

Figure 4 shows the morphologies of ceramic glaze surfaces observed using TTSEM. It was clearly seen that cracks reduced with the increasing of temperature. It showed that at 800°C, the glaze was not matured and resulted in an uneven melted glaze. As temperature increased from 900 to 1100°C, the glaze melted with a smooth surface. However, at 900°C, cracks appeared on the surface of the glaze and the cracks began to heal as temperature rose from 1000 to 1100°C. A flat, smooth layer with no waves glaze can be obtained when the glaze received an adequate fusibility in the condition of applied heat treatment [15].

Effects of different soaking times

The effects of soaking time on the surface of the glaze were studied. As shown in Figure 5, cracks reduced as the soaking time increased. The morphology of the glaze surface sintered at 900°C with 4 hours of soaking time and analyzed by TTSEM showed a smooth surface as shown in Figure 4. A proper fusion of the raw materials can be achieved by prolonging the soaking time [16] and a longer soaking time causes the crystals to partially or completely melt, resulting a smoother and glossier surface.

Soaking the sample at the desired melting temperature for a period of time is to ensure the maturity of clay and give the glaze an opportunity to flow freely and heal imperfections thus producing a flawless glaze [17], especially for coarser particles that produce gases during decomposition and leaving defects on the surface of glazes. It can be claimed that 900°C sintered glaze with 4 hours soaking time able to produce a smoother glaze with no defect as compared to 1, 2, and 3 hours of soaking.

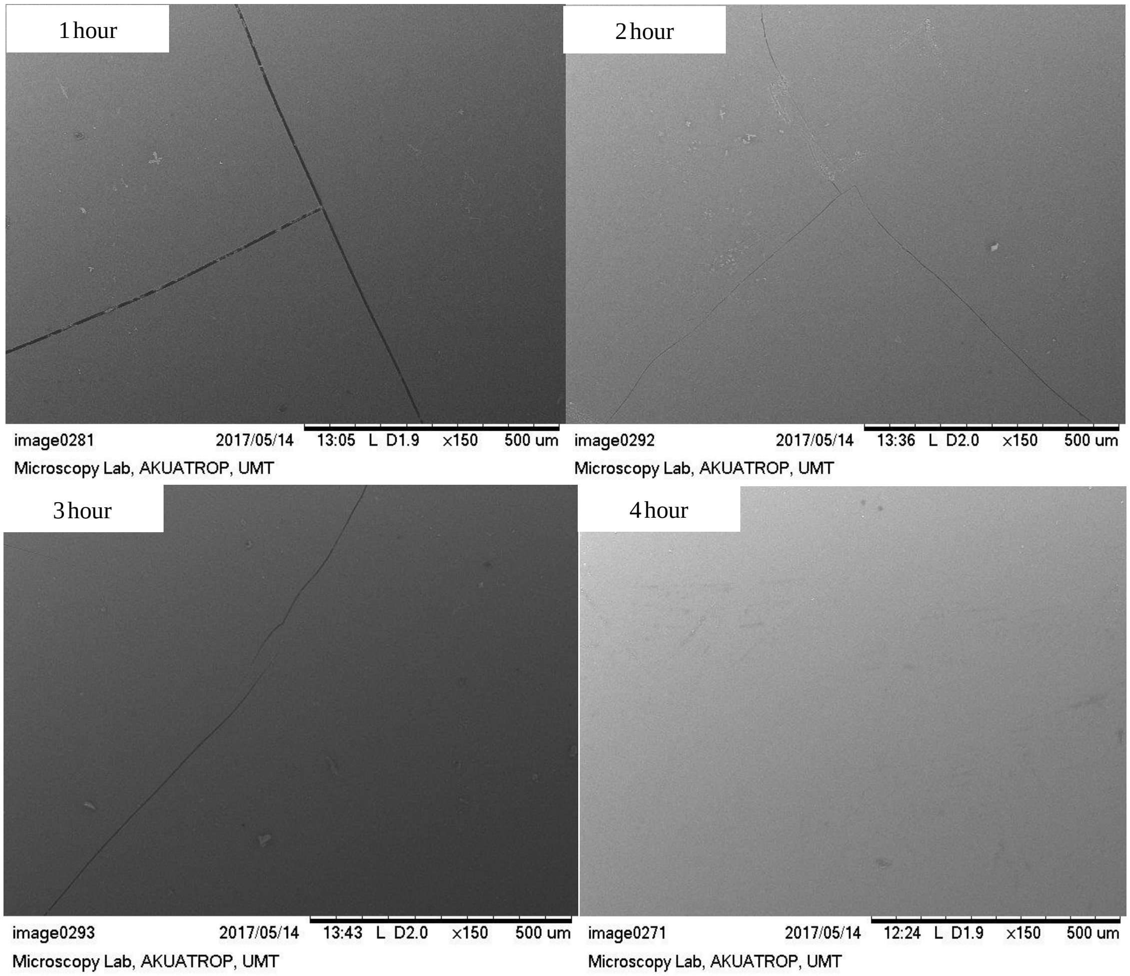

Figure 5 SEM images of the glaze melted at 900°C and soaked at different soaking times

CONCLUSION

The morphology and hardness of the ceramic glaze changed with melting cycles. Addition of secondary flux helped lower the melting point of the glaze where the glaze was able to melt at 900C. The observations on the soaked glaze at different soaking times indicated that the cracks present were able to heal completely as the soaking time was increased to 4 hours. Increase in sintering temperature also hardened the glaze from 92.5 to 93.7 *hv*. It can be concluded that the lowest temperature to melt the glaze is at the temperature of 900°C with 4 hours of soaking time.

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