



Study on Particle Size Distribution and It's Effects on Shrinkage, Porosity and Bulk Density of Tri-Axial Porcelain Tiles

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ABSTRACT

Soft porcelain possess very specific physico-chemical characteristics to play an important role in the domestic and industrial. Formulations of soft porcelain were studied in the different laboratories and production simulation of tile making processes were compared with the standards. Different samples of ceramic raw materials A1, A2, A3 and B were prepared by grinding processes for different time periods. The samples were characterized, their particle size and composition influence were evaluated. After that the tri-axial porcelain tiles A1/T, A2/T, A3/T and B/T were prepared. Results reveal that on decreasing the particle size, a negligible change was observed in fired linear shrinkage. The microstructure of porcelain has a decisive influence on the mechanical and functional behavior of products. The fired ceramic results can be incorporated into porcelain stoneware tiles production as economic, technological and environmental correct solution. Bulk density from 2.10 – 2.59 gm/cm³, water absorption 0.56 – 1.11 %, apparent porosity 2.08 – 3.11 % and fired linear shrinkage 13 – 14 % were observed. The results show that compositions containing a higher amount of quartzitic clays possess lower shrinkage in the temperature range of 1050 – 1150 °C. Tri-axial body compositions containing higher amount of china clays showed the lowest water absorption and higher strength due to better densification.

KEYWORDS

China clay, mullitization, soda feldspar, linear shrinkage, porosity

INTRODUCTION

Porcelain tiles fundamentally display a tri-axial formulation, based on three raw materials clay, feldspar and quartz, which contribute the necessary plasticity for forming in green state. This traditional ceramic formulations consist generally of Tri-axial components i.e. of at least three components that play the three fundamental roles for optimum processing and hence performance of the final products, china clay for plasticity, feldspar for fluxing and quartz as filler for the structure. Formulations of tri-axial porcelain usually involve 45 wt. % of plastic component, 30 wt. % soda feldspar and 25 wt. % quartz used for soft porcelain. After suitable production processing, these formulations should lead to high strength performing porcelains with high frost, chemical resistances and low porosity. The typical microstructure of porcelain ceramics consist of a glassy matrix that contains mullite and dispersed partially undissolved quartz particles. Commercial feldspar and quartz for porcelain contains a maximum of 0.3 wt. % iron oxide as the major impurity. Various researchers have studied the effect on various physical properties viz water absorption, bulk density, modulus of rupture, porosity, mullitization, shrinkage and whiteness on tri axial porcelain tiles [1-2]. The chemical composition of clay also effects the rheological properties of the ceramic paste which play an important role in slip casting. The most frequent techniques were used for the manufacture of white ware porcelain articles [3]. The phenomenon of mullitisation is also influenced by the grade of feldspar which particularly effects the formation of secondary mullite. The quality of feldspar is determined by the amount of orthoclase and albite in the soda feldspar, the most commonly used fluxes in porcelain composition [4]. Now a days, mixed feldspars are considered suitable fluxing agents for porcelain manufacture, since they develop a very viscous glassy phase that embeds the new forming crystals and a part of the residual crystals present in the microstructure that enhance the densification process. Secondly the mixed feldspar minimizes the effect of changes in mineralogically mining. In this investigation, a chemical approach was applied on soft porcelain compositions with soda feldspar as fluxing agent to study. In porcelain tiles bodies, porosity is an undesirable result of the vitrification process of the ceramic body and adversely effects

the physical and chemical properties of the ceramic materials. The different firing process leads to big differences in the percentage of crystalline phases in the end products. Mullite and glass constitute the major phases of porcelain. Quartz is more abundant than mullite. Most of the reactions occurring during firing are kinetically governed processes that do not reach thermodynamic equilibrium in porcelain stoneware production as the industrial cycles are less than 1 hour. Thus because of fast firing process, porcelain stoneware became a non equilibrium material. The high technological properties of porcelain stoneware, especially water absorption, chemical and frost resistance, bending strength and abrasion resistance cause highest increase in production and sales over all other ceramic building material [5-6].

MATERIALS AND METHODS

Location of the samples: The lumps of china clay samples taken from Merta city, Nagaur, Rajasthan. The main utility of ball clay for preparation of ceramic bodies with the high bonding qualities and tensile strength lies in its plasticity. Therefore, it is used in blend with to improve workability and strength of bodies on drying. The big lumps of soda feldspar and quartz samples collected from village Paota; Jaipur; Rajasthan and Kekri; District Ajmer respectively.

Raw materials characterization: The clays were washed and pulverized into micronized fine powder ready for batching. These raw materials were procured in micronized form with grain size of 300 nm maximum. Particle size distribution was evaluated using particle size analysis Make: CILAS model no. 1064. The chemical analyses of all raw materials were observed. Gravimetric method was used to determine LOI and SiO₂ whereas Al₂O₃, CaO and MgO were determined volumetrically. Fe₂O₃ and TiO₂ detected by spectro-photometric and Na₂O and K₂O were determined by flame photometer [7]. The ceramic raw materials of porcelain tiles i.e. china clay with LOI - 13.93 %, SiO₂ - 46.30 %, Al₂O₃ - 35.2 %, CaO - 1.78 %, MgO - 0.28 %, Na₂O - 0.10 %, K₂O - 0.29 %, TiO₂ - 0.105 %, Fe₂O₃ - 0.088 % and Li₂O found nil with purity of 99.97 %. Feldspar with LOI - 0.32 %, SiO₂ - 70.75 %, Al₂O₃ - 14.11 %, Na₂O - 11.86 %, K₂O - 2.14 %, Fe₂O₃

00.58, LiO₂, TiO₂, CaO and MgO found nil with purity of 99.76 %. The chemical analysis of quartz samples observed that SiO₂ – 99.99 % and Fe₂O₃ – 00.01 % with purity of 100 % were used to prepare tri-axial porcelain tiles.

The water content at which a reduction in water content will not cause a decrease in volume of the clay mass but an increase in water will increase the volume is known as shrinkage limit of clays. Dry linear shrinkage of clay is determined by measuring the decrease in one dimension of a plastic weight upon drying and expressing it as percentage on plastic basis. Dry shrinkage is also an important property of clay by which the particle size maintained. The shrinkage depends upon the plasticity of clay which also indicates the warping tendency during drying. Linear shrinkage was determined as per the IS 4589:2002 (III revision) standard specification of Bureau of Indian Standards [7]. Porcelain ceramic tiles prepared with a liquid binder or water. All fine particles of ceramic raw materials having a high shrinkage during drying process followed by firing, the resultant problems of warping and distortion may occur. Therefore, the plastic components in the porcelain ceramic tiles reduced the ultimate defects due to high shrinkage. The dry linear shrinkage was calculated as follow [7-9].

$$\text{Dry Linear Shrinkage (\%)} = \frac{L_p - L_d}{L_p} \times 100$$

Where,

L_p = distance in mm between the two reference points on the plastic weight.

L_d = distance in mm between the two reference points on the dry weight.

Fired Shrinkage: After taking the dried specimens prepared for dry linear shrinkage and measuring the dry length, fire the same specimens at the required firing temperature. Removing the specimens after cooling and measure the distance between the reference points. Fired linear shrinkage expressed as a percentage of fired length on fired basis. The sintering process develops glassy phase and progressively closes the capillaries that form the apparent porosity. The closed pores is known as fine closed porosity, with a more and less spherical morphology structure from the closing of the apparent porosity [10]. On the other hand in the series of large isolated pores is distinguish, with a spherical shape and that size usually exceed 10 μm, known as coarse porosity. This pore type is found in those regions that have been generated from the feldspar and is the cause of the greater or lesser stain resistance of unglazed porcelain. When the fired porcelain body polished, the closed porosity is exposed in the surface and dirt can then lodge in these voids [11]. The formation of porcelain tiles at low temperature of a fused phase with low viscosity from the flux facilitates pore expansion and the formation of spherical porosity of greater size [12-13].

RESULTS AND DISCUSSION

Mullite (3Al₂O₃·2SiO₂) is considered the principal crystalline phase in porcelains and responsible for their microstructure and mechanical properties. The formation of mullite depends mainly on the type and proportion of china clay used. Pure and well-crystallized china clay provide better results of mullitisation at above 1000 °C, while disordered china clay forms mullite at higher temperature i.e. above 1200 °C. There is competition between the formation of mullite and crystallization of the amorphous silica present in the tri-axial during firing. Moreover, the proportion and nature of the quartz used in the composition of porcelain batch influences the proportion of un-reacted residual quartz during firing. Quartz possesses a higher coefficient of thermal expansion as compared to that of the surrounding glassy phase, hence giving rise to thermal stresses, which remarkably affect the strength of porcelain. Different authors have demonstrated that mullitisation can be increased by catalytic ions such as Fe³⁺ and Ti⁴⁺. These metallic ions help in mullite formation by replacing the Al³⁺ ions in the glass structure during firing. The presence of small amounts of Fe³⁺ and Ti⁴⁺ in clay modifies the chemical composition of the ceramic bodies and therefore the sintering behav-

ior which the case of porcelain characterized by mullitisation [14].

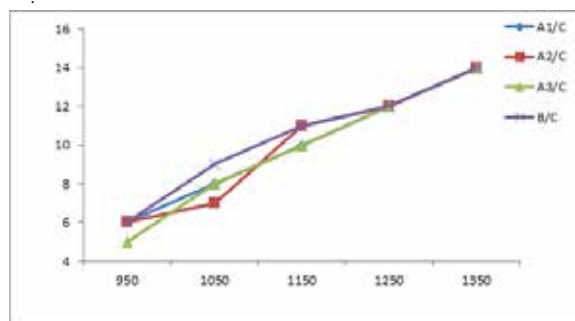


Fig. 1. Effect of firing temperature on fired linear shrinkage of china clay samples.

Fired linear shrinkage of selected china clay A1/C, A2/C, A3/C and B/C samples are less than 16%, the maximum requirement of fired linear shrinkage were obtained as per Bureau of Indian specification. The observed values of fired linear shrinkage of all selected china clay samples, lies between 13 to 14 % at temperature 1350 °C. No significant change in the results of fired linear shrinkage was observed by decreasing the particle size of minerals shown in figure 1. It was observed that linear shrinkage increase with the increase in temperature.

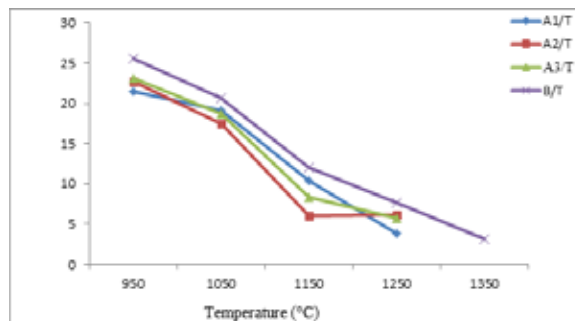


Fig. 2. Apparent porosity of porcelain tiles at different temperatures.

The apparent porosity for the porcelain tiles made from the composition of china clay, feldspar and quartz i.e. A1/T, A2/T, A3/T and B/T were found to increase with firing temperature as shown in figure 2. The increase in apparent porosity at higher firing temperature is caused by bloating, which takes place as gasses are expelled from the matrixes and causes the increase in porosity. Porosity of the tiles expected to decrease with increase of clay content in the tri-axial composition. This is because when clay particle is heated produces glassy phase that fills the pores and decrease the porosity. The tile with lowest apparent porosity has the lowest water of absorption, high bulk density and high compressive strength.

The porosity of the fired sample is associated to other physical properties such as linear shrinkage and water absorption. The properties are function of firing temperature from 950 to 1350 °C. Total porosity depicts a similar trend that linear shrinkage, which initially increases, reaches to a maximum value and decreases after 1150 °C due to close porosity increasing. Finally, as water absorption is directly related to open porosity, its value decreases in the overall temperature range. To discuss the degree of densification, bulk density rather than absolute density, must be compared because the content of crystalline phases effect absolute density. It has been observed that bulk density has a similar behavior to linear shrinkage i.e. initial increase is followed by an abrupt decrease in the bulk density as shown in figure 3.

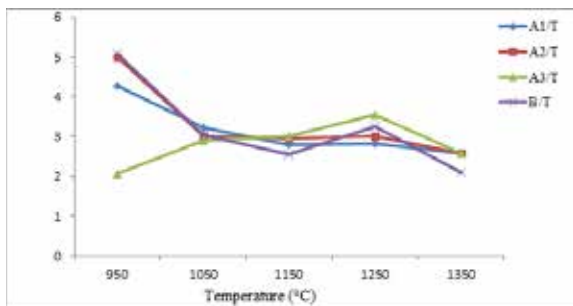


Fig. 3. Bulk Density of porcelain tiles at different temperatures.

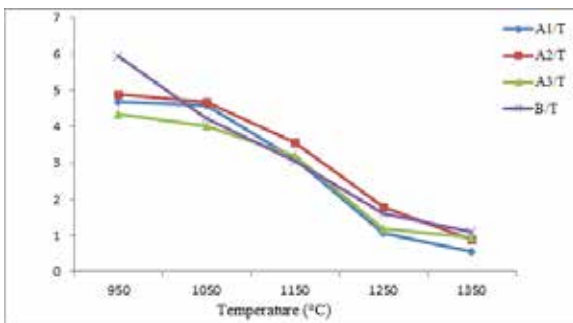


Fig. 4. Water Absorption of porcelain tiles at different temperatures.

Water absorption decreases with increases in firing temperature due to the formation of the liquid phase and densification at high firing temperature shown in figure 4. On firing, these properties show the typical trend of a ceramic body sintered by a viscous liquid phase mechanism. In fact, it is apparent that open porosity decreases with increasing firing temperature due to the formation of a glassy phase that is mainly originated from the feldspar. Because of the surface energy forces created by the fine pores of ceramic body, liquid phase tends to approach the particles and therefore open porosity decrease [15]. Combination of both open and closed porosity gives rise to the total porosity decrease in the first steps of sintering, reaching a minimum value at 1150 °C and then followed by increase. The similar behavior was observed in almost all porcelain bodies.

CONCLUSION

The composition of raw materials to the suitable firing temperature for the preparation of porcelain tiles was observed. Raw material of the suitable firing temperature for the preparation of porcelain tiles was clearly observed and concluded that tile made from the composition have the water absorption of 0.56 – 1.11 %, bulk density of 2.10 – 2.59 gm/cm³, apparent porosity of 2.08 – 3.11 % and linear shrinkage of 13 – 14 %. Analysis shown that coarse closed porosity and interparticle porosity are the important properties. To improve the microstructure of porcelain tile bodies more efforts needed to focus on decreasing porosity. Minerals that generates these types of pores i.e. feldspar and quartz, it is necessary to control their physical, chemical and fired characteristics. To avoid of diminish the porosity other components will be used. These results are consistent with reports in the literature and confirm that the use of larger particles increases the bulk density of the green compact. This is due to the more efficient packing of this type of particle inside the granules during the granulation stage [16]. The decrease in the degree of milling of feldspar causes an increase in the average size of the particles, that will make the granules of the materials. Although the degree of densification of the green compact is higher in these conditions.

The possible solution is to introduce a flux at high temperature to allow the clay components to react at low temperature, that helps to closing of the fine residual closed porosity and which would generate in addition a fused phase of high viscosity that avoid the increase in size of coarse closed porosity. The flux must be properly milled, since the smaller is grain size larger the interparticle contact area. Hence the greater will be the sintering in the solid phase and the working temperature will be lower.

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