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The effect of particle size of body components on the processing parameters of semi transparent porcelain

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ABSTRACT

The effect of the particle size of quartz and K- feldspar on the sintering behaviour and technical properties of semi transparent porcelain were investigated. This study was implemented by “the Factorial Experimental Design Method” to determine the effect of factors on semi vitreous porcelain production. The sintering behaviour of the samples were examined by using an optical dilatometer. The phases and their respective amounts in the microstructure were determined by way of Rietvelt X- ray diffraction (XRD) technique. Furthermore, the values such as shrinkage, water absorption, bulk density, porosity, thermal expansion, light transmission measurements were taken on the samples and the microstructures were studied by SEM. It was observed that as the particle size of quartz and K- feldspar decreased; the viscosity of the liquid phase fell down, the relative amount of secondary mullite crystals increased and the large pores were removed among the particles and the densification rate increased. Data were analysed by Minitab 13.20 software, and assessed in relation to the amount of glassy phase and its viscosity. The effect of K- feldspar particle size on the densification of porcelain is less than the change of heat treatment and quartz particle size.

1. Introduction

Porcelain is a glassy bonded semi-transparent material with a white colour and low water absorption. In general, a porcelain body is typically made of clay, feldspar and quartz along with other constituents to promote the desired characteristics [1,2]. Because of the complex interplay between raw materials, processing routes and kinetics of the firing process, porcelains represent some of the most complicated ceramic systems. Because of the complex reactions, obtained microstructure controls the final properties of porcelain such as bulk density, porosity, water absorption, strength, deformation, transparency and colour [3–5].

The microstructure of porcelain consists predominantly of mullite, undissolved quartz, glass, and even, rarely, residual feldspar and pores. Mullite starts to form between ~ 1050 and 1200 °C, and its transformation is completed in the temperature range of 1200 – 1400 °C under conventional firing conditions [1, 2, and 6]. The mullite level is proposed to be dependent on the solubility of Al_2O_3 in the glass phase,

measured to be $1.19 (\pm 0.1)$ moles of alumina per mole of flux ($R_2O + RO$) [7,8]. Dissolution rate of quartz in glass matrix is a function of firing cycle but relatively insignificant below 1200 °C. If the quartz grains are very fine, they are highly dissolved in the matrix, whereas if grains are too coarse, they induce crack generation. The effect of quartz dissolution in glass matrix on the mechanical and physical properties were studied by many researchers but still there is an argument regarding the positive or negative effect of residual quartz especially on porcelain strength [9–12]. The glassy phase formed during firing process fills the pores in the microstructure and enables the intensification of the interior. The formation of viscous liquid starts at different temperatures depending on the chemical composition of the porcelain body. During the firing of a typical porcelain, feldspar particles react with SiO_2 from Meta kaolin to form an eutectic liquid at 990 °C for potassium feldspar and 1050 °C for sodium feldspar. The glassy phase is placed in the particle boundaries at 1200 – 1400 °C [13,14].

An intensive structure can be provided when the proportion of the open pores is reduced to 0.5%. [15]. The density of the porcelain is

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Table 1
Factors and the levels of the experiments.

Factors	Levels			
	I	II	III	IV
Quartz	Quartz 101	Quartz 102	Quartz 103	–
K-Feldspar	K- feldspar 101	K- feldspar 102	K- feldspar 103	
Temperature (°C)	1150	1175	1200	1250

Table 2
Experimental heat treatment parameters.

Heating rate (K min ⁻¹)	Dewell temperature (°C)	Dwell time (Minutes)
10	1150	30
10	1175	30
10	1200	30
10	1250	30

Table 3
Chemical analysis of raw materials.

	SiO ₂	Al ₂ O ₃	Fe ₂ O ₃	TiO ₂	Na ₂ O	K ₂ O	CaO	MgO	L.O.I.
Kaolin	49.65	35.86	0.07	0.01	0.09	1.96	0.08	0.23	12.05
Albite	70.11	18.28	0.10	0.17	10.05	0.3	0.64	0.12	0.18
K- Feldspar	68.30	17.02	0.16	0.47	2.24	10.81	0.16	0.33	0.51
Quartz	98.44	0.92	0.05	0.02	0.16	0.11	0.13	–	0.17

Table 4
Particle size results of the raw materials.

	d(0.1) μ m	d(0.5) μ m	d(0.97) μ m
Quartz 101	1.52	4.25	12.16
Quartz 102	3.79	21.18	67.16
Quartz 103	10.12	36.19	141.10
K-Feldspar 101	1.49	3.97	13.99
K-Feldspar 102	1.93	13.51	50.16
K-Feldspar 103	4.58	29.18	82.19

Table 5
Chemical analysis of the semi transparent porcelain body composition.

	Na ₂ O	MgO	Al ₂ O ₃	SiO ₂	K ₂ O	CaO	TiO ₂	Fe ₂ O ₃	LOI
%	1.15	0.15	22.74	65.52	4.57	0.40	0.09	0.41	4.97

increased by increasing the temperature due to the elimination of open pores by viscous flow. Viscous flow is controlled by the melt viscosity. The melt viscosity is directly related to the composition and the particle size of the body. A high density structure can be achieved by increasing

Table 6
The component contents of the compositions.

Composition code	T1011	T1012	T1013	T1021	T1022	T1023	T1031	T1032	T1033
Quartz 101	x			x			x		
Quartz 102		x			x			x	
Quartz 103			x			x			x
K Feldspar 101	x			x			x		
K Feldspar 102		x			x			x	
K Feldspar 103			x			x			x
Albite	x	x	x	x	x	x	x	x	x
Kaolin	x	x	x	x	x	x	x	x	x

the ratio of the submicron particles. Since the powers of fine-sized particles are higher than those of large-sized ones, their mobility is also higher. Thus, they can react more easily and increase the speed of sintering [15–20].

Another important effect of the particle size of the porcelain composition is the rheology of the porcelain slurry suspension and the packing density of the structure to be formed at the end of the shaping process. Particle packing is directly controlled by the particle size distribution of the material being processed. For this reason, particle packing is important to all particulate/fluid systems. The diffusion distance of the particles can be shortened to increase the sintering speed with a dense packing [21–23]

A factorial design is a research method that includes two or more factors. A factor is an independent variable in an experiment, especially those that include two or more independent variables. One advantage of a factorial design is that it creates a more realistic situation, which can be obtained by examining a single factor. In addition, it uses a notation system that identifies both the number of factors and the number of values or levels that exist for each factor. [24]. In the study of Bondioli et al., they investigated the mathematical model of the pressing pressure for porcelain tile production, the effect of the maximum firing temperature and the duration of the firing period on the firing contraction. Two-level multi-factorial experiment design was used to model the factors affecting fired shrinkage. The results show that it is possible to define the effect of different factors on the reduction of firing temperature with a limited number of tests. [25].

In this study, the bodies were prepared by using quartz and K-Feldspar in three different particle size distributions in a fixed porcelain body composition and passed through four different firing cycles. The technical and physical development of bodies during and after sintering process have been investigated. Experimental studies were carried out based on the factorial experiment design (the order of construction and repetition depends on the rules). Considering the significant effect of the temperature on the test results, 3²4¹ general factorial experiment was designed. The analysis of the experiments was implemented using Minitab 13.20 package program.

2. Experimental studies

The factors and levels determined in the experiments are given in Table 1. Test specimens were fired in a laboratory type Forno Ceramica kiln at 1150, 1175, 1200 and 1250 °C. Experimental heat treatment parameters are given in Table 2. The chemical analysis results of the raw materials were determined by the fluorescence method (XLab 2000-Spectro) and results are given in Table 3.

The particle size distribution of quartz, K- feldspar and albite were measured by laser diffraction method using Malvern Master Sizer 200 G model and the results are given in Table 4. The theory based chemical analysis of the standard recipe used for every sample is given in Table 5. The component contents of the compositions were given in Table 6.

To prepare the batches, raw materials were dispersed in an aqueous

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