

Porous single-fired wall tile bodies: Influence of quartz particle size on tile properties

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Received 12 March 2009; received in revised form 26 June 2009; accepted 3 August 2009

Available online 31 August 2009

Abstract

This study examines the effect of quartz particle size in raw material composition customarily used for the manufacture of porous single-fired wall tile bodies on the characteristics of the green tiles and on the thermal and mechanical properties of the fired tiles. Quartz particle size was varied, while the quantity and particle size of the other raw materials were kept constant. Tile compacts were formed by uniaxial pressing and fired at different peak temperatures. The resulting fired microstructure was then characterised and tile thermal and mechanical properties were determined. Microcrack formation around quartz particles leads to hysteresis of the coefficient of thermal expansion during heating and cooling. The studied mechanical and thermal properties are shown to be a function of the magnitude of the hysteresis and porosity. This relationship is independent of the operating variables (pressing pressure, operating temperature, and quartz particle size) used. The results obtained confirm that the green and fired properties of porous single-fired wall tiles may be considerably enhanced, while holding low shrinkage and high porosity, compatible with low moisture expansion, by reducing quartz particle size and appropriately adjusting the pressing pressure and peak firing temperature. This should enable thin and/or large-sized porous wall tiles to be manufactured, without (immediate or delayed) curvatures, and with a higher breaking load than that required by the standards.

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Keywords: Thermal expansion; Mechanical properties; Porosity; Final microstructure; Traditional ceramics

1. Introduction

Annual wall tile production currently stands at more than $4 \times 10^8 \text{ m}^2$ in Europe, with turnover exceeding $3 \times 10^9 \text{ €}$ /annum. The greatest demand in wall tiles in recent years has been for thin, large-sized, white-firing earthenware tiles. For the manufacture, by single firing, of these thin glazed sheets with high porosity (water absorption $\geq 15\%$), without immediate or delayed curvatures, and with an appropriate breaking load, it is essential, among other factors, that the tiles should have good (green and fired) mechanical properties.

The raw materials mixture contains a balanced proportion of plastic materials (clays) and non-plastic materials (calcium carbonate, quartz, or feldspathic sands), which are milled to a particle size that allows green tiles to be pressed with appropriate green mechanical strength, whose constituents will react in the short firing cycles used in industrial practice^{1–4} ($\approx 40 \text{ min}$).

Calcium carbonate needs to have a small particle size in order to decompose rapidly and completely during the first stages of the firing cycle, while the resulting calcium oxide must fully react with clay minerals decomposition products to produce calcium aluminosilicates and/or calcium magnesium aluminosilicates^{5–7} (if calcium magnesium carbonate is used). Pinholes would otherwise develop in the glazed surface and/or the end product would contain amorphous phases, or even residual calcium oxide, which could hydrate, damaging the tiles^{8–11} (leading to delayed curvatures, crazing, etc.). Quartz is a cheap lightweight raw material, a relatively large quantity of which (35–50 wt%) is needed in the body composition, partly as siliceous clays (quartz content of 15–30 wt%) and partly as feldspathic sands (quartz content above 60 wt%), mainly for two reasons. First, because quartz is the principal determining component of the high mean coefficient of thermal expansion of the resulting tile, which is required in order to obtain an appropriate glaze-body fit. Secondly, because quartz acts as a filler and contributes to dimensional stability by reducing firing shrinkage.

The effect of quartz¹² (grain size and content) on the mechanical properties of porous wall tile has received little attention,

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especially in regard to particle size. This is in sharp contrast with the abundant information available on porcelain, vitreous china sanitary ware, and porcelain tile. For these materials, with abundant glassy phase and low porosity, the principal factor influencing strength is considered to be quartz particle size, as demonstrated repeatedly by several researchers.^{13–18} This effect is basically caused by two simultaneously acting mechanisms related to the difference between the coefficients of thermal expansion of the quartz and of the glassy matrix: on the one hand, the difference in thermal expansion between both materials has a strengthening effect, by subjecting the glassy matrix to microscopic residual compressive stress; on the other, the magnitude of the stresses produces cracks around the quartz particles when quartz particle size exceeds a critical size, a_c , thereby causing partial stress relaxation and increasing microstructural damage, which may even lead to particle detachment. This microstructural damage adversely affects the product's mechanical properties.

In porous wall tile bodies, whose microstructure and composition differ entirely from those of porcelain bodies (higher porosity and smaller glassy phase content), the foregoing two mechanisms also need to be considered, albeit in a different measure. Thus, the presence of cracks around and within the large quartz particles has also been observed.¹²

One way of estimating the extent to which microcracking develops during tile cooling in the kiln is by determining the hysteresis of the coefficient of thermal expansion around 573 °C during heating and cooling of a fired test piece in a dilatometer.^{19,20} The magnitude of the hysteresis of the thermal expansion has been shown to determine the thermal and mechanical properties in anisotropic and/or composite materials.^{21–24} This hysteresis effect between heating and cooling, observed for these sintered materials, has also been attributed to the occurrence and recombination of microcracks.

This study examines the effect of quartz particle size in raw material composition customarily used for the manufacture of porous single-fired wall tile bodies on the characteristics of the green tiles and on the thermal and mechanical properties of the fired tiles. Quartz particle size was varied, while the quantity and particle size of the other raw materials were kept constant. Tile compacts were formed by uniaxial pressing and fired at different peak temperatures. The resulting fired microstructure was then characterised and tile thermal and mechanical properties were determined.

This study has two objectives. The first is to determine the effect of quartz particle size on the mechanical and thermal properties of the fired body, and to attempt to relate these properties to the fired tile's main characteristics (porosity, phase content, microstructure, etc.). Indeed, determining how microcrack density, estimated by thermal expansion hysteresis, affects the above properties constitutes an important new approach. The second objective is to explore the possibility of improving fired tile mechanical strength, reducing quartz particle size and appropriately modifying the operating variables, while holding or improving the other key characteristics and properties of the semi-processed and the finished product. The purpose of the foregoing is to enable thin and/or large-sized porous wall tiles

Table 1
Raw materials chemical composition.

wt%	E-clay	S-clay	Quartz	Calcite	K-feldspar
SiO ₂	63.2	64.0	98.7	0.2	68.7
TiO ₂	1.3	1.01	0.08	–	0.03
Al ₂ O ₃	23.8	20.2	0.54	0.1	17.3
Fe ₂ O ₃	1.2	2.35	0.05	0.05	0.14
MgO	0.4	0.36	0.01	0.2	0.05
CaO	0.3	0.55	0.01	55.7	0.44
Na ₂ O	0.3	0.17	0.04	–	2.20
K ₂ O	2.6	2.09	0.32	0.01	10.61
l.o.i.	6.8	6.60	0.25	43.5	0.52

to be made, without (immediate or delayed) curvatures, with a higher breaking load than that required by the standards.

2. Materials and procedure

The following raw materials were used: an English ball clay, E-clay (HSM clay from Imerys Tiles Minerals), a white clay from Teruel (Spain), S-clay (Roma clay from Minera Sabater, containing mainly kaolinite, micaceous material, and free quartz); calcite (from Zaera, Calaf); potassium feldspar (FK-100 from Incusa, also containing sodium feldspar); and three quartz sands from Sibelco of the same composition (containing traces of potassium feldspar), but having a different particle size distribution (PSD): F-quartz, M-quartz, and C-quartz, for fine, medium and coarse quartz particle sizes, respectively. Apart from the English ball clay, the raw materials were from Spain and are widely used in manufacturing white-body earthenware tiles.

The chemical composition of the raw materials is given in Table 1. The corresponding particle size distributions are detailed in Table 2.

Three different slips were prepared consisting of 45% S-clay, 10% E-clay, 15% calcite, 5% K-feldspar, and 25% quartz of different grain size.

Slurries were prepared by dispersion with a high-speed disperser. Slurry loading was 65 wt%, deflocculant content being 0.5 wt% of a sodium metasilicate–sodium tripolyphosphate mixture in a 3:1 ratio. Stirring time was 1 h. Slurry viscosity was measured as a function of shear rate between 10^{−1} and 10³ s^{−1}.²⁵

The suspensions were spray dried at 200 °C in a laboratory spray dryer. Prior to compaction, the moisture content of the

Table 2
Cumulative particle size distribution.

	Particle size (µm)				
	<10%	<25%	<50%	<75%	<100%
E-clay	–	–	0.5	3.5	21
S-clay	–	–	1.1	6.0	20
Calcite	2.4	4.7	9.1	17.4	28.5
K-feldspar	1.5	3.5	10.9	23.0	35.5
C-quartz	3.9	35.3	41.8	83.4	129
M-quartz	2.5	8.3	23.0	44.3	67.3
F-quartz	2.4	4.6	7.9	14.1	23.1

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