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Effect of reduction of thickness on microstructure and properties of porcelain stoneware tiles

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Abstract

Porcelain stoneware tiles with reduced thickness have a smaller mass to be fired, thereby reducing energy consumption during firing and saving raw materials. However, the relationship between tiles with reduced thickness, their microstructure and technical properties is unclear. The present work deals with the analysis of the microstructure and properties of porcelain stoneware related to the reduction of the tile thickness. Tiles were manufactured from an industrial spray-dried powder batch, and pressed in lab-scale with varying thicknesses from 1.5 to 5.5 mm. Compacting pressure of 39.2 MPa was held constant and the maximum firing temperatures varied from 1180 to 1220 °C. At 1180 °C the samples were not completely densified corresponding to higher water absorption values (> 3%) and lower modules of rupture (< 45 MPa). When sintered at higher temperatures, the products were more robust in terms of variability of physical and mechanical properties. Higher fracture strength was reached for tiles fired at 1220 °C. However, samples fired at 1200 °C presented the highest bulk densities (\sim 2300 kg/m³) and water absorption around 0.3%, regardless of the thickness. In all cases, the modules of rupture were above the required standardized values (> 35 MPa). Nevertheless, the minimum required breaking loads for wall and floor tiles were only reached for thicknesses larger than 2.5 and 4.5 mm, respectively.

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1. Introduction

Porcelain stoneware tiles are building materials with outstanding technical properties such as mechanical strength, wear and chemical resistance. In the last decade, the global production of porcelain stoneware increased markedly when compared to other ceramic tiles. In fact, the technical properties of porcelain stoneware, coupled with improved esthetic appearance, gave it a prominent role in the tile market [1]. Porcelain stoneware tiles are characterized by low values of water absorption (<0.5%), according to ISO 13006 [2], which is an essential feature, making

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them frost resistant and therefore available for outdoor flooring and wall cladding in cold climates [3].

The production of tiles with reduced thickness is an important technological innovation aimed at decreasing both the production costs per unit surface area and the costs of packaging and transport [4]. Thin porcelain stoneware tiles (down to 3 mm) have very low specific weight (as low as 7 kg/m²) compared to conventional 9 mm tiles, which are around 21 kg/m^2 . Therefore, thickness reduction can save costs of production, logistics and transport, furthermore reducing raw materials consumption. Moreover, large tiles with low thickness show certain degree of flexibility, making them novel building and construction materials [5]. Some applications include floorings mounted over previous paving, ventilated façades tunnel coverings, insulating paneling, indoor furniture

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(table tops, doors), self-cleaning and antibacterial surfaces or support for photovoltaic ceramic panels [6-8].

The industrial processing of porcelain stoneware includes three main stages: (a) wet milling and homogenization of raw materials, followed by spray-drying of the resulting suspension; (b) uniaxial pressing at 35–45 MPa of the spray-dried powder with a moisture content of 5–7 wt%; (c) fast firing for 40–60 min at 1180–1220 °C to obtain maximum densification [3].

The manufacturing of tiles with reduced thickness is generally based on uniaxial pressing. For medium or small size products (up to $450 \times 450 \text{ mm}^2$), the raw materials can be shaped using conventional pressing devices using the drop hammer system [9]. For larger tiles (e.g., $1000 \times 3000 \text{ mm}^2$ or $1200 \times 3600 \text{ mm}^2$), an alternative pressing technology was proposed by Gozzi et al. [10], which applies the screw press principle. In this process, ceramic thin sheets ($\sim 3 \text{ mm}$ thick) can be produced with high apparent dry density values ($1.88-2.03 \text{ g/cm}^3$) that correspond to a low total green porosity (23-29%). Da Silva et al. [11] introduced tape casting as an alternative process for shaping porcelain tiles with thickness around 2 mm. For tape cast tiles, sintering reactions occur at lower temperatures, mechanical strength is higher and water absorption is lower when compared to pressed tiles with the same thickness.

Nevertheless, the influence of process parameters on microstructure and properties of thin porcelain stoneware tiles has not been studied so far. Therefore, the objective of this work is to investigate the effect of the tile thickness reduction on the microstructure and physical properties of porcelain stoneware produced from an industrial powder batch, uniaxially pressed in lab-scale and fired at different temperatures.

2. Experimental procedure

2.1. Industrial processing

Raw materials were processed in a ceramic tile industry through milling and spray-drying. The batch of porcelain stoneware tile was composed of two clays, feldspar, kaolin and talc. All experiments were performed in samples prepared from the same batch. The chemical analysis of the raw materials was carried out by X-ray fluorescence (Philips PW 2400) and is presented in Table 1, corresponding to a typical composition of porcelain stoneware currently produced in a tile manufacturing plant located in Santa Catarina State, Brazil. Milling of raw materials was performed during 11 h in a discontinuous ball mill (11,400 L inner volume, high-alumina coating and grinding balls). The slip presented 4.3 wt% particles retained in a 325 ASTM mesh sieve ($45 \mu m$). Sodium silicate (15 wt% Na₂O/32 wt% SiO₂, 1580 kg/m³) was used as a dispersant. The slip density was determined by pycnometry (100 mL) and the slip viscosity was measured in a viscometer (Brookfield, RVDVII, shear rate of 20 rpm). The particle size distribution of the slip was determined by laser diffraction (CILAS 1064), after dispersion in water using ultra-sound (60 s) to avoid agglomeration of the particles.

After milling, the slip was discharged into an underground tank with 40 t capacity. During the discharge process, a vibrating sieve (60 ASTM mesh) was used for separation of undesirable particles. A binder (0.5 wt% polyethylene glycol, Tenacer, Zschimmer& Schwarz, 1210 kg/m³, pH 7.2), was added into the tank and the slip was stirred during 24 h for complete homogenization.

After homogenization, the slip was granulated in an industrial spray-dryer at countercurrent flow. The spray-dried granules were stored during 24 h in an 80 t silo. The moisture content of the granules was assessed using a moisture meter (Ohaus, MB35 Halogen) and the particle size distribution was measured by sieving, using a vibrational system with 35, 50, 100 and 200 ASTM mesh sieves during 5 min at 60 Hz vibration. About 150 kg of the spray-dried granules were taken apart to be used in the pressing process.

2.2. Lab-scale processing

The laboratory step was comprised by pressing, drying and firing of the tiles. Pressing was performed in a semi-automatic hydraulic press (Gabbrielli Sesto Fiorentino) at 39.2 MPa. The tiles were pressed into five different thicknesses, 1.5, 2.5, 3.5, 4.5 and 5.5 mm. An arithmetic mean for every property measured at different levels was obtained from 20 specimens.

Bulk density of the green bodies was determined by the Archimedes' principle. The ceramic tiles were dried at 110 ± 10 °C for 24 h and fired in a continuous roller kiln (Nassetti) at maximum firing temperatures of 1180, 1200 and 1220 °C with heating and cooling rates of 40 °C/min, and a total of 60 min from cold to cold.

Table 1 Chemical analysis (XRF) of raw materials, and batch composition of porcelain stoneware tiles.

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Composition	wt%	SiO_2	Al_2O_3	Na ₂ O	K_2O	MgO	CaO	TiO_2	Fe_2O_3	LoI
Raw materials										
Feldspar	30.8	72.8	13.9	7.3	0.7	0.9	1.9	0.1	0.2	2.1
Clay A	26.0	49.7	27.0	2.5	0.9	0.7	7.9	0.1	1.0	10.1
Clay B	4.0	83.8	10.8	< 0.1	0.4	0.1	0.2	0.2	0.3	4.2
Talc	8.0	73.0	2.5	-	0.1	18.4	0.2	0.2	1.2	4.4
Kaolin	30.4	68.7	20.1	1.2	3.9	0.1	0.1	0.1	0.8	5.0
Spray-dried powder										
* * *	100.0	66.2	18.8	3.2	1.7	1.3	2.9	0.1	0.6	5.2

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