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Microstructure and technological properties of porcelain stoneware tiles moulded at different pressures and thicknesses

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Abstract

Moulding pressure and thickness are pre-fixed variables in the porcelain stoneware tile industry. This paper examines the effect of both parameters on technological and microstructure properties of unfired and fired porcelain stoneware bodies. The moulding pressure in unfired tiles has a noticeable effect on bending strength and load borne. Common technological properties such as water absorption, porosity, bulk density and bending strength were obtained from the fired tiles. The results indicate that the variation in properties is independent of the thicknesses of the tiles. Moulding pressure affects at lower values, whereas higher moulding pressure produces tiles with similar technological properties. The microstructure exhibits a constant amount of mullite and quartz in all pieces after the firing process. Observations from scanning electron microscopy show that mullite crystals enlarge as moulding pressure increases, but the shape of the crystals are unaffected by the individual thicknesses. Furthermore, the aspect ratio of the mullite needles increases with the moulding pressure, which is invariable to the thicknesses of the tiles. Although the total porosity remains constant, the number of pores is influenced by the thickness. The moulding pressure also influences pore size. The results show a strong relationship between the number of closed pores and the thickness of the ceramic tile.

Keywords: B. Porosity; D. Mullite; Porcelain stoneware; Fast-firing; Density

1. Introduction

Porcelain stoneware tile is a ceramic material that exhibits superior technical performance in the construction sector. It offers many advantages (lower water absorption and porosity and higher flexural strength) in the design of pavements or coverings compared with other ceramic tiles, such as glazed stoneware. Porcelain stoneware is composed of clay, flux agent and filler. The clay is typically comprised of kaolinite, which confers plasticity to green paste and is the precursor of mullite crystals [1–4]. The fluxing agent is feldspar and the filler is quartz, which most likely lead to higher strengths of the unfired tiles [5,6]. Firing bodies containing these three components exhibit a grain and bond microstructure, which consists of coarse quartz grains joined by a finer bond or matrix that contains mullite crystals and a glassy phase [7].

The variables that govern the industrial process include moulding pressure, firing temperature and soaking time. Data on the last two variables, which have been extensively researched, are reported elsewhere. Regarding moulding pressure, unfired tiles can be shaped by a single- or double-pressing procedure. When single pressing is applied, pressure values in the range of 30–50 MPa are used, which are the values typically presented in the literature [8]. Abadir et al. [9] studied the effects of moulding pressure (35–55 MPa), firing temperature and soaking time in porcelain tiles; however, no previous studies exist in which higher pressures are employed.

Its relatively high density may limit the use of porcelain stoneware in innovative applications, such as for the covering of internal walls or the manufacturing of ventilated facades. Recent studies have reported the manufacture of lightweight porcelain stoneware with reduced densities through the addition of foaming agents, such as CeO_2 [10] or SiC [11]. However, the foaming agents cause a reduction in mechanical strength, which limits the maximum attainable reduction in weight. The latest market trends are progressing towards large dimensions (i.e., $60 \times 60\ cm^2$ or $120 \times 60\ cm^2$) and a reduced tile thickness (3–6 mm) [12,13]. However, strong technological limitations constrain the fabrication process. Raimondo

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et al. [14] examined large-sized ceramic slabs that were manufactured through an innovative ceramic process, including an assemblage of a ceramic fibreglass composite.

Information about the effect of thickness on the technological properties of porcelain stoneware tiles is scarce. The most common technological properties of tiles are assumed unaffected by their thickness; thus, the study of their effect on the microstructure of fired materials has been disregarded.

The present study extends a previous research [8] that concluded that moulding pressure affects the shape of mullite needles, which have a direct effect on the bending strength of porcelain stoneware tiles. The aim of this present study is to expand the previous results by establishing the influence of thickness and moulding pressure on the technological properties and microstructure of typical porcelain stoneware tiles. The microstructural study focuses on the morphology of mullite crystals because the technological properties of the tiles are dependent on the formation of these crystals.

2. Experimental

2.1. Materials and methods

To obtain the tiles, 50% kaolinitic clay (EuroArce), 40% feldspar (Rio Pirón) and 10% quartz sand were mixed in a planetary mill with distilled water (1:1) for 30 min. Based on the process by Martín-Marquez et al. [15], porcelain stoneware tiles (55 mm × 15 mm) were moulded in a manual hydraulic press to get four thicknesses (3, 4.5, 6 and 9 mm). Five different moulding pressures (20, 40, 60, 80 and 100 MPa) for each thickness were applied during 1 min. Higher moulding pressures were not utilised because Fahrenholtz [16] indicated that moulding pressures above 100 MPa may lead to the development of pressure gradients and other defects that can affect the quality of the tiles after pressing and firing. A specific tile thickness was maintained at different pressures by controlling the quantity of ceramic powder tucked into the mould. The experimental work was divided into two parts: the first part entails the study of the unfired tile properties, such as bulk density and bending strength. The second part entails the microstructural and technological characterisation of the bodies fired at 1200 °C after a fast-firing process, which is similar to the procedure commonly used in industrial manufacturing of porcelain stoneware tiles.

By employing the same procedure described in Pérez et al. [8], the bulk density was measured in a hygroscopic balance after weighting the sample in air and immersing it in distillate water.

The bending strength, σ_f, of 10 test pieces was measured using a three-point loading test in an electronic universal tester (Servosis), with a crosshead speed of 0.1 mm/min for unfired pieces and 1 mm/min for fired pieces (UNE-EN 843-1).

The linear shrinkage, LS (%), of the fired samples was determined by the following equation:

$$LS = \frac{L_s - L_c}{L_s} 100 \quad (1)$$

where L_s and L_c are the length (mm) of the unfired and fired specimens, respectively. The linear shrinkage values of the 10 specimens were averaged for each firing temperature.

The determination of water absorption (WA, %), bulk density (BD, g/cm^3) and apparent porosity was achieved under conditions found in ASTM C373-88. The test was performed on four representative specimens for each temperature. The unfired samples were measured after protecting them with a waterproof coating (commercial varnish) to avoid the collapse during the experiment.

The open porosity, ε_0 (%), total porosity of the sample, ε_T (%), and closed porosity, ε_c (%) were measured following the ASTM C329-88 standard.

The microstructure of the fired specimens was examined by field-emission scanning electron microscopy (FESEM) in a Hitachi S-4800 microscope using an acceleration voltage of 20 kV. The porosity of polished surfaces was evaluated using Esprit 1.9 image analysis software. For the analysis of phase assemblages and morphology, fresh fracture surfaces were etched for 4 min in 15% HF solution, washed ultrasonically with distilled water and ethylic alcohol, dried and subsequently coated with Au–Pd in a Balzers SCD 050 sputter. Secondary

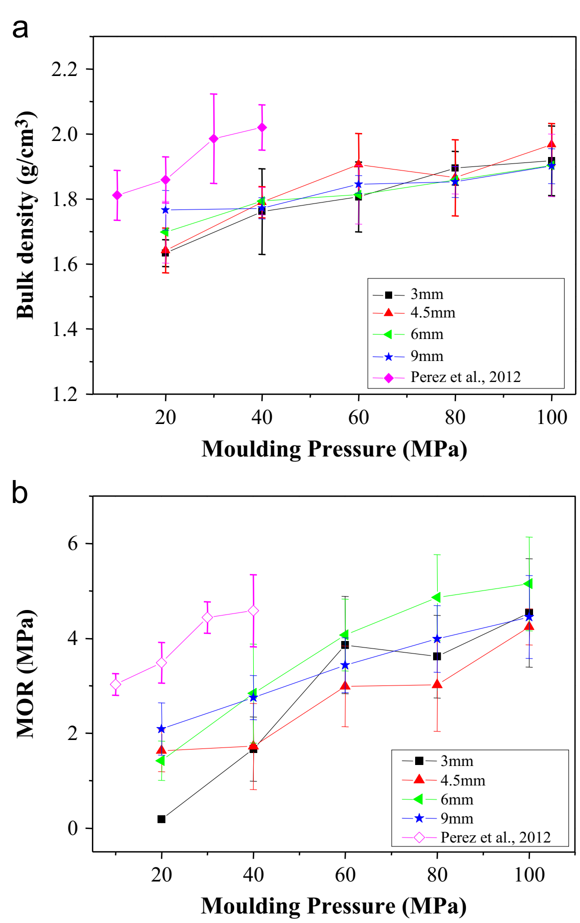

Fig. 1. (a) Bulk density of unfired tiles shaped at different moulding pressures and thicknesses and (b) variation of bending strength of unfired tiles with moulding pressure. Diamonds data are from Pérez et al. [8] (both straight and dotted lines drawn in Figs. 1–4, 69 and 12 are included for visual guidance).