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**Pyroplastic Behaviour of Porcelain Stoneware Tiles:  
Comparison of the Wet and Dry Manufacturing Routes**

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**ABSTRACT**

The growing concern on the economic and environmental sustainability of ceramic tile production is drawing attention to the dry manufacturing route as alternative to the wet cycle conventionally used for porcelain stoneware tiles. At the same time, the control of firing deformations, connected with pyroplasticity, turned to be crucial with development of large size, rectangular shapes, low thickness, and with use of energetic fluxes and micronized fillers. The present work merges these two issues with the objective of comparing the pyroplastic behaviour of industrial-like body compositions processed by both the wet and the dry routes. Pyroplasticity is active when sintering acts through viscous flow of an abundant amorphous phase, as typical of porcelain stoneware. It is thought to depend on microstructural variables, such as amount, size, shape and mutual arrangement of coarse grains, and on the viscosity of the liquid phase formed at high temperature as well. Four batches were prepared in two different ways: by ball milling using water and deflocculant and by ball milling using dried raw materials. Then granulation was carried out in sieves (wet) or microgranulation (dry). The laboratory simulation of the industrial processing was carefully carried out in order to get every couple of bodies (wet route vs. dry route) with the same chemical and mineralogical composition, and with minimal differences in terms of particles size distribution. Fired samples were characterized by determining the water absorption, bulk density, open and closed porosity, phase composition (XRD-Rietveld) and microstructure (SEM with image analysis). A pyroplasticity index (PI) was determined by three-points flexural test at temperature of maximum densification. Results reveal a conspicuous difference in the pyroplastic behavior between bodies manufactured by the wet route ( $7.4-9.5 \cdot 10^{-5} \text{ cm}^{-1}$ ) and the dry route ( $4.4-6.1 \cdot 10^{-5} \text{ cm}^{-1}$ ). The PI values are strongly affected by the number and volumetric fraction of skeleton grains ( $>5 \mu\text{m}$  in size, mean diameter  $13-17 \mu\text{m}$ ) consisting essentially of quartz. In fact, the amount of coarse-grained quartz is higher in dry processed tiles ( $700-900 \text{ per mm}^{-2}$ ,  $13-20\% \text{ vol}$ ) with respect to the tiles manufactured by the wet route ( $300-600 \text{ per mm}^{-2}$ ,  $7-13\% \text{ vol}$ ). No clear correlation arose between PI and the amount of vitreous phase, whose variations are moderate ( $62-70\% \text{ wet vs. } 68-72\% \text{ dry}$ ). The different microstructure obtained by the wet and the dry routes has repercussion on the chemical composition of the vitreous phase and the crystalline load of the viscous matrix, which both are expected to affect the pyroplastic behaviour. The amount of crystalline phases in the viscous phase is  $6-16\%$  (wet) and  $<3\%$  (dry) thus it seems not to play a key role with respect to skeleton grains. The PI values correlate almost linearly with the viscosity of the vitreous phase at the temperature of maximum densification rate, but only for the batches manufactured by the wet route. In conclusion, pyroplasticity appears to be a complex behavior depending on both microstructure (particularly number, volume and mean distance of skeleton grains) and vitreous phase features (chemical composition and viscosity at high temperature). The sintering of porcelain stoneware tiles proved to proceed by different paths in the dry and the wet routes, thus originating different microstructures, which in turn affect the firing deformations.

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