A NEW DESIGN OF GREEN AND FIRED MICROSTRUCTURE IN THE PRODUCTION OF CERAMIC WALL TILES

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The fast-firing process in ceramic wall tile production demands a specific approach regarding the design of the raw mineral composition. This demand is related to standard EN 159 BIII (water absorption (10% by mass), which includes the regulation of warpage and centre curvature values. The characteristics of ceramic powders [1] and the behaviour of particle contacts during the compaction process [2] provide the basis for obtaining the desired green microstructure of ceramic tiles. The green microstructure of ceramic tiles has a strong effect on the behaviour of these systems during the sintering process, presenting also the start of the specific "defect" in the microstructure of the final product [3]. Understanding the role of all the specified phenomena is important for making improvements in the quality of ceramic wall tiles obtained in the fast firing process.

The main physical characteristics of the industrial products (ceramic wall tiles) were controlled in the plant for a period of two months. The most pronounced warpage phenomenon, concerning all the dies (D1-D6) of the press, was statistically observed in the case of the end dies (specifically D1), the central part being preferable. The paper correlates the density of green products, compaction degree of the spray-dried powder and mineral composition of the raw components in order to define the causes of distortion phenomena and to keep them within the standard limits.

[1] FONTANA, F.; GROSSI, A.; MOLORI, P.G. *Study concerning flow distribution inside a spray drier,* Ceramica Acta, 1, 15-23 (1999)

[2] Walker, W.J.; Reed, I.S. *Granule fracture during dry pressing,* Am.Ceram.Soc.Bull., 28(6), 53-57(1999)

[3] RANOGAJEC, J.; PANI, J.; MARINKOVI-NEDUIN, R. *Compaction rate diagrams of multicomponent atomized ceramic powder,* Ceram.Eng.Sci. Proc. 14(11-12), 32-42(1993)

EXPERIMENTAL

• *Industrial raw material components*: domestic (based on illite and carbonates) and commercial (based on kaolinite) clayey material, mixed with ground ceramic roofing tile, which acts as a filler.

• *Ceramic spray-dried powder*: average ceramic spray-dried powder and powders sampling from the specified part of a defined industrial die (middle and end die). Fractions obtained by sieving procedure (Table 1).

• *Ceramic wall tiles*: green ceramic tiles, corner and central parts (working pressure 220, 250 and 270 bar); freshly sintered and aged 2 months, warpage values: corner (+0.1 to +0.7) and central part (-0.6 to -1.4).

Methods: standard chemical analysis; XRD-phase analysis *(Philips Pw 1050 CuK_{α})*; textural analysis-low temperature nitrogen adsorption *(ASAP 2000, Micrometrics*, pores up to d=300 nm); thermal characteristics *(Thermomicroscope Leitz IA up to 1500°C)*; microstructure *(SEM, JOEL, ISM-35).*

RESULTS AND DISCUSSION

Chemical analysis: *domestic clayey* material is poor in clay minerals, but very rich in carbonates and quartz; *commercial clayey material* has a higher content of Al_2O_3 and a significant content of K_2O (2,42 % by mass), giving a glass phase with a high coefficient of viscosity.

Thermal characteristics: *commercial clayey material* - high temperature of shrinkage (944 °C) and softening onset (>1300°C), causing improper glass phase properties; *domestic clayey material* - shrinkage onset 838°C, softening 1211°C, considerably earlier than the commercial material; *industrial raw material batch* - shrinkage onset 891°C, obviously more determined by the commercial component and too late with regard to the industrial kiln (1120°C); softening 1237°C, more influenced by the domestic component, resulting in improper microstructure.

Textural investigations (pore size distribution-PSD, surface area-SA) were performed in order to correlate: *working pressure, position of the die* in the press (total 6), *particular part of the green tile* (corner and central part). Pressure of 250 bar can be characterized as optimal, yielding more uniform pore size distribution and surface area among the tile segments and die position in the press, Fig.1. Unstable distribution of pores identified in the corner part, strongly influenced by the applied pressure, is absent in the middle part of the same tile.

Microstructure investigation: *ceramic wall tiles aged two months* (corner and central part) - transformation of gehlenite into anorthite is not finished; preferable morphology of cluster of anorthite form is not present, the secondary CaO phase predominating, particularly intensified in the case of the corner part/ D1 die; *fresh wall ceramic tile* (corner part) - separation of the crystalline phase from glass phase was initiated, caused by unfinished transformation of gehlenite into anorthite phase; gaps between ex-spray-dried granules were identified. The sintering process was intensified inside the granules more than between them. The worst situation was in the case of the end die (D1) at p=270 bar.

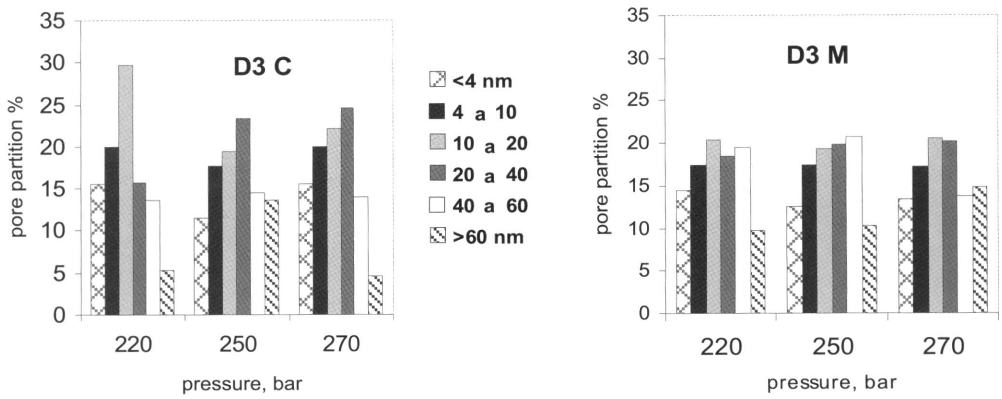

Figure 1. Pore size distribution for corner (C) and middle (M) part of the middle die (D3)

The particle size distributions of the spray-dried powder in the end and middle die in comparison to the average initial spray-dried powder, Table 1, give additional data for explaining the observed differences. The disarrangement of particle size distribution is obvious in end die D1of the press, particularly noticeable for B and D fractions. For the first two pressures (220 and 250 bar) there is no significant difference in surface area of tile middle and corner part in contrary to the highest applied pressure (270 bar). Evidently, a considerable amount of energy, up to p=270 bar, is wasted in order to obtain satisfactory results.

Fraction		Initial powder		End die (D1)		Middle die (D3)	
Ref.	Size μm	PSD mass%	SA m^2/g	PSD mass%	SA m^2/g	PSD mass%	SA m^2/g
A B (A+B)	> 500 500-355	3.5 30.3 33.8	14.68	2.7 26.7 29.4	15.37	3.0 30.0 33.0	15.33
C D (C+D)	355-250 250-125	38.1 23.5 61.6	15.95	38.4 26.3 64.7	21.80	38.5 23.4 61.9	14.22
E F (E+F)	125-90 < 90	2.7 1.1 3.8	15.54	4.0 1.2 5.2	14.30	3.1 1.1 4.2	15.57
Moisture, % by mass		4.0		4.3		4.0	

Table 1. Particle size distribution and surface area of average powder samples.

CONCLUSIONS

The selection of the optimal pressure for obtaining a proper initial green structure depends not only on the raw material composition, but also on the specific pressure unit in the industrial line. The positions of the die have to be taken into account, the end-die is particularly critical with regard to potential distortion phenomena. Pore size distribution and surface area of the tile segments can be a guide-line in obtaining an equilibrium state, bringing about a stable microstructure in the final product, keeping a longer period of usage.