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Forming of thin porcelain tiles: A comparison between tape casting and dry pressing

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Abstract

Thin porcelain tiles (down to 3 mm thickness) are currently made by dry pressing, while thin advanced ceramics (down to 0.1 mm thickness) use tape casting as forming step. This study proposes an alternative way of manufacturing thin porcelain tiles by tape casting. A systematic comparison between dry pressing and tape casting was made for a 2 mm thick tile fabrication. A current industrial formulation with the same particle size distribution and green density was employed, and the sintering temperature was varied in the range of 1180–1220 °C with heating rate of 40 °C/min. Firing shrinkage, loss on ignition, mechanical strength, and water absorption were measured and the microstructure after sintering was analyzed. The results showed that tape cast tiles are more homogenous and therefore presented higher values of mechanical properties.

Keywords: A. Pressing; A. Tape casting; Porcelain tiles; Thickness

1. Introduction

Forming processes turn a powder mix, plastic material or slip into a defined shape. There are many processes available to perform this function [1]. Some of these may be classified as traditional, namely uniaxial pressing, extrusion or slip casting. Other manufacturing techniques, such as isostatic pressing, injection molding, are rather associated to advanced or high-tech forming processes [2,3].

An important technological innovation for porcelain tiles is the reduction of the thickness. The advantages are the lowering both the costs of production per unit of surface area and the costs of packaging and transport [4]. The search for novel applications has not only required investigation of new facilities but the design of new products, such as porcelain tile laminates, which may be up to 4 m long and just 3 mm thick [5]. This innovation focused initially at replacing materials such as wood, plastic, or metal in applications so far inaccessible to ceramics, such as an armored doors or kitchen shelves, since the possible tile sizes that can be made are practically unlimited. However, the manufacturing processes involved in fabricating those products have more recently focused on shaping thicker porcelain tile preforms, which are subsequently cut into standard tiles sizes in order to provide a more flexible manufacturing process in comparison to the current powder pressing techniques [5].

Currently thin porcelain tiles are normally produced by dry pressing. This method is the simultaneous compaction and shaping of a powder or granular material confined in a rigid die or flexible mold. For industrial pressing operations, powder feed is in the form of granules of controlled size and deformability. Presses used for dry forming of tiles are generally hydraulic, whose force is generated by oil under pressure, which acts upon the piston of a cylinder [6].

Thin advanced ceramic are processed by tape casting with film thickness varying from 100 to 1500 μm [7–19]. Most of cast tapes are based on Al_2O_3, but SiC, ZrO_2 and glass ceramics have also been reported.

Tape casting is a well-established technique for large-scale fabrication of ceramic substrates and multilayered structures. Slurry consisting of the ceramic powder in a solvent, with addition of dispersants, binders and plasticizers, is cast onto a

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stationary or moving surface [20]. Multilayered capacitors or packages, piezoelectric, fuel cells, and lithium ion batteries might be produced by this technique. Tape casting is specially indicated for manufacturing large, thin and flat ceramic parts. It is comparable to traditional slip casting as it also uses a fluid suspension of ceramic particles as the starting point for processing [2].

The tape is formed when the slurry flows on a moving carrier polymeric film, and is then dried. Thin sheets of ceramic may also be formed by pouring the slurry onto a flat surface and moving a blade over the surface to form the film. The powder concentration in the slurry must be very reproducible and the viscosity of the slurry feed must be well controlled. Temperature has a significant effect on the viscosity and should be controlled [6].

This work proposes to use tape casting for manufacturing thin porcelain tiles in comparison to the traditional dry pressing process. In both cases, the effect of maximum firing temperature on microstructure and physical parameters was investigated.

2. Experimental procedure

2.1. Industrial step

The porcelain tile batch was prepared in industrial scale by wet milling and spray drying. The porcelain tile mix was composed by two clays, kaolin, feldspar and talc. The chemical analysis of the raw materials was determined by X-ray fluorescence (Philips PW 2400). Milling of the raw materials was performed during 11 h in a discontinuous ball mill (11400 L inner volume, with high-alumina coating and grinding balls). The slip presented 4.3 wt% particles retained in a 325 ASTM mesh sieve. Sodium silicate (15 wt% Na_2O, and 32 wt% SiO_2; 1580 kg/m^3) was used as a dispersant. The density of the slip was determined by pycnometry and viscosity was measured by a Brookfield viscometer (Model RVDVII, shear rate of 20 rpm). The particle size distribution of the slip was determined by laser diffraction (CILAS model 1064) with the sample dispersed in water using ultra-sound (60 s) to avoid agglomeration.

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/m^3, pH 7.2), was added in the tank and the slip was stirred during 24 h for complete homogenization. Then, 100 kg of slip was taken apart for the tape casting process. After homogenization, the slip was spray-dried. The spray-dried powder was stored during 24 h in an 80 t silo. The moisture content of the powder was determined using a moisture meter (Ohaus, MB35 Halogen) and the granulometric distribution was measured by sieving, using a vibrational system with 35, 50, 100 and 200 ASTM mesh sieves during 5 min at 60 Hz. A certain mass (150 kg) of the spray-dried powder was separated to be used in the pressing process.

2.2. Laboratory step

Forming, drying and firing of the porcelain tiles were carried out in laboratory scale. Two forming processes were applied: dry pressing and tape casting. In both the final thickness of formed tiles was $\sim$2 mm. Pressing was performed in a semi-automatic hydraulic press (Gabbrielli Sesto Fiorentino) with 12.3 MPa of pressure. The pressed samples after drying presented approximately the same bulk density of the samples shaped by tape casting.

The doctor blade of the tape caster was adjusted to define the thickness of the casting film. The deposition on a plastic film was performed manually. The samples were dried at room temperature for 24 h and then cut into 30×100 mm^2 specimens, which corresponds to the approximate dimensions of the porcelain shaped by dry pressing.

Due to the high amount of water used during the forming of the porcelain by tape casting, it was necessary to use a plasticizer (5 wt% plasticizer) that minimize the impacts of drying shrinkage, reducing the probability of cracks.

All properties values at different levels were obtained as the arithmetic mean of 20 specimens.

The specimens formed by pressing were dried in a drying chamber at 110 ± 10 °C for 24 h, while the specimens shaped by tape casting were dried at room temperature (25 °C) for 6 days, followed by 24 h of drying in a drying chamber at 110 ± 10 °C.

Bulk density of green bodies was characterized by the Archimedes principle. The samples were fired in a continuous roller kiln (Nassetti, Italy) at maximum firing temperatures of 1180, 1200 and 1220 °C with heating and cooling rates of 40 °C/min.

2.3. Characterization of fired tiles

The linear shrinkage and loss on ignition of the ceramic tiles were determined after firing. The linear shrinkage was determined using a caliper, and the loss on ignition by the calcination method (at each temperature and firing cycle). The mechanical strength was measured by the three-loading method (Gabbrielli Sesto Fiorentino, Italy) according to ISO 10545 [21]. The water absorption by the boiling water method was also performed following ISO 10545 [22]. The microstructural analysis was carried out on the fractured surface of the samples using a Scanning Electron Microscope (SEM, Philips XL30, Netherlands). All results were subjected to an analysis of variance (ANOVA) [23].

3. Results and discussion

3.1. Characterization of the porcelain batch

Table 1 shows the composition of the formulation and the chemical analysis of the raw materials used in the porcelain tile batch. Feldspar A presents a low amount of Fe_2O_3 and TiO_2, resulting in a white color after firing. The Na_2O content reduces the firing temperature of the formulation. Clay A shows a higher

Table 1
Chemical analysis (wt%, XRF) of raw materials.

Raw material	Formulation	Al_2O_3	CaO	Fe_2O_3	K_2O	MgO	Na_2O	SiO_2	TiO_2	LoI
Feldspar A	30.8	13.9	1.9	0.2	0.7	0.9	7.3	72.8	0.1	2.1
Clay A	26.0	27.0	7.9	1.0	0.9	0.7	2.5	49.7	0.1	10.1
Clay B	4.0	10.8	0.2	0.3	0.4	0.1	< 0.1	83.8	0.2	4.2
Talc	8.0	2.5	0.2	1.2	0.1	18.4	–	73.0	0.2	4.4
Kaolin	30.4	20.1	0.1	0.8	3.9	0.1	1.2	68.7	0.1	5.0
Sodium silicate	0.8	–	–	–	–	–	–	–	–	–
Plasticizer	0.5	–	–	–	–	–	–	–	–	–
Total	100.0	18.8	2.9	0.6	1.7	1.3	3.2	66.2	0.1	5.2

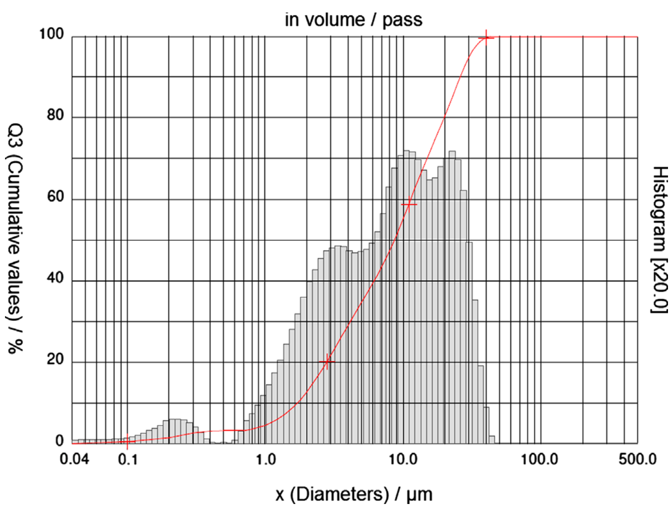

Fig. 1. Particle size distribution of the porcelain tile batch.

amount of Fe_2O_3 and therefore a darker color. The high Al_2O_3 content results in a higher firing temperature. Clay B is basically composed of silica and alumina, being complementary in the formulation of the porcelain mixture. Talc is used in the formulation to reduce the viscosity of the liquid phase formed during the firing of the ceramic formulation. Finally, the kaolin presents K_2O and Na_2O reducing the refractoriness.

The viscosity, density and particle size distribution of the slip were also determined. The viscosity and the density of the slip used in spray-dryer were 500 mPa s and 1610 kg/m^3, respectively, which are standard values for the porcelain tile production. The best viscosity found in preliminary tests to provide a uniform shaping at tape casting was 6030 mPa•s at 20 rpm, so this value was used, which corresponds to a density of 1710 kg/m^3.

The particle size distribution of the powder batch is shown in Fig. 1.

The moisture content of the spray-dried granules was 6.2 wt %. The size distribution of the spray-dried powder is shown in Table 2. The weight percentages refer to the fraction of the spray-dried powder retained in the sieves.

A suitable packaging during pressing is the result of an adequate flow and size distribution of the ceramic powder. Spray-dried powders smaller than 125 µm are less fluid during pressing. Powders with higher fluidity are those in the range of 125–500 µm [24], which corresponds to the spray-dried powder used is this work.

Table 2
Size distribution of the spray-dried powder.

Sieve ASTM mesh (µm)	#35 (500)	#50 (297)	#100 (149)	#200 (74)	< 74 µm
Retained (wt%)	39.7	50.7	7.9	0.7	0.4

3.2. *Characterization of the porcelain tiles*

Bulk density (green and after firing), firing shrinkage, loss on ignition, mechanical strength, water absorption of the compacts were measured; the microstructure was also observed. All results were analyzed using ANOVA with $p < 0.05$.

3.2.1. *Bulk density and green microstructure*

During pressing, the initial volume of the compact is given by the sum of the volume of solid particles and the empty spaces between them. The empty spaces are pores, and there are basically two types of pores that make up the microstructure of the green compact: intragranular pores (spaces between the particles that comprise the granule) and intergranular pores (set of voids that form during packing of the granules) [25,26]. For the tape casting process, the initial volume is given by the sum of the volume of solid particles and the water restrained in the body. As the water is evaporated during drying, the bulk density after drying is given by the sum of the volume of solid particles and the empty spaces between them, left by the evaporation of water. Fig. 2 shows the green microstructure of the porcelain tile formed by pressing and tape casting. In the porcelain tiles formed by pressing it can be observed that inside the body there are granules and intergranular pores. On the other hand, porcelain tiles formed by tape casting do not show intergranular pores; their microstructure is apparently more homogeneous and the pores are probably caused by water evaporation during drying.

Bulk density can influence other properties of the ceramic tiles during firing, such as firing shrinkage, mechanical strength and water absorption. Table 3 shows the mean values of those properties, with no significant differences between samples.

3.2.2. *Firing shrinkage*

Distinct behaviors can be observed for firing shrinkage as a function of the forming method and firing temperature (Fig. 3). For materials fired at 1180 °C, the firing shrinkage was higher

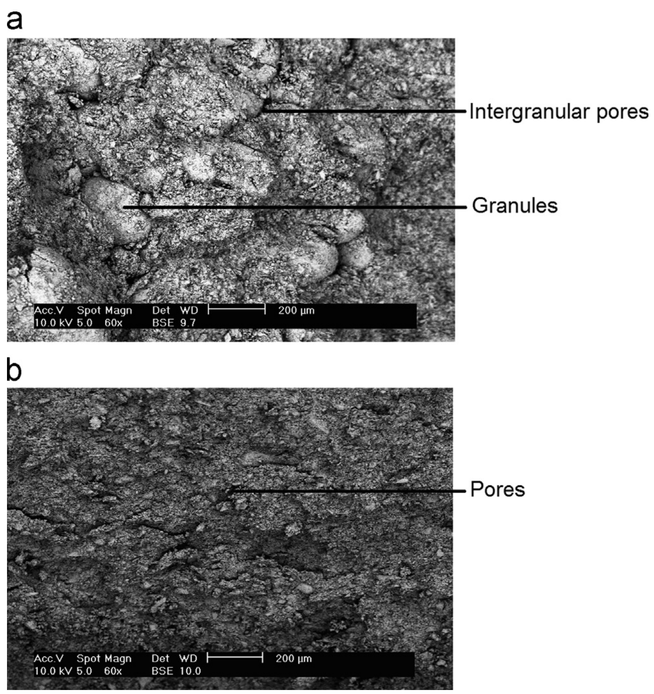

Fig. 2. Green microstructure of the porcelain tiles: (a) Pressing; (b) Tape Casting.

Table 3
Bulk density of the tiles formed by tape casting and dry pressing.

	Tape casting	Dry pressing
Bulk density (kg/m^3)	1620	1621
Standard deviation (%)	0.6	1.0

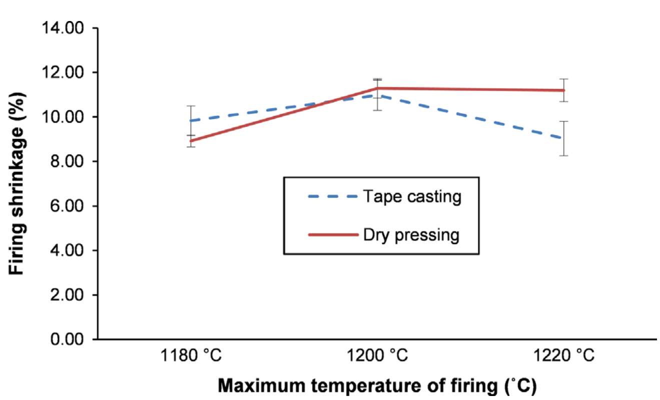

Fig. 3. Firing shrinkage of the porcelain tiles formed by tape casting and dry pressing.

using tape casting. At 1200 °C there is no significant difference between the means and at 1220 °C firing shrinkage was higher for the pressed tiles.

The higher firing shrinkage at lower temperature for the tape casting method can be attributed to the best packing obtained due to the favorable flow of the particles in a liquid media. The homogeneity of the green bodies shaped by tape casting enhances

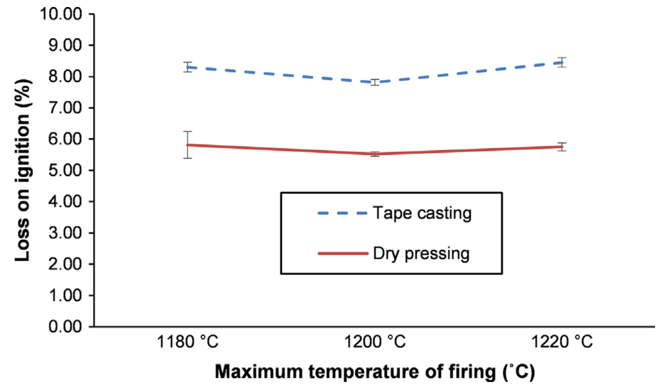

Fig. 4. Loss on ignition of the porcelain tiles formed by tape casting and dry pressing.

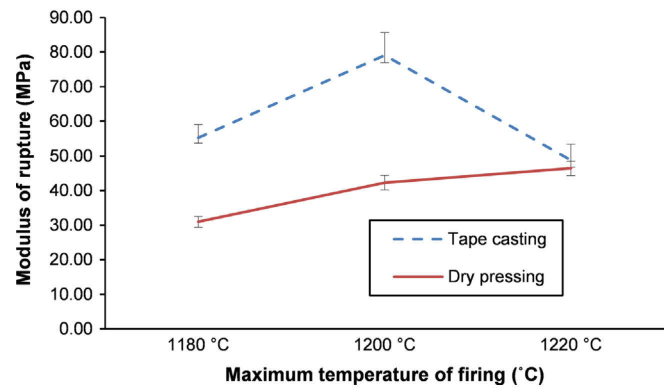

Fig. 5. Mechanical strength of the porcelain tiles formed by tape casting and dry pressing.

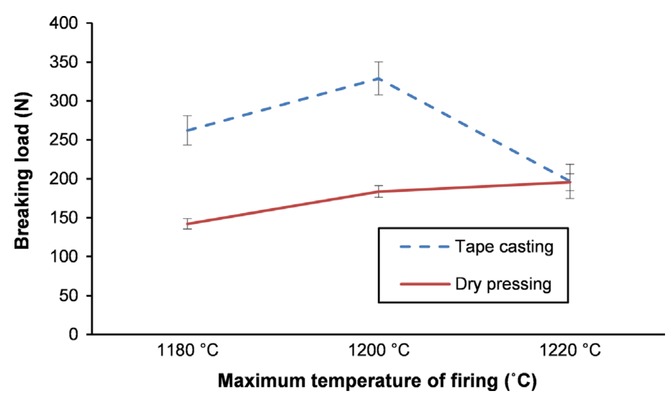

Fig. 6. Breaking load of the porcelain tiles formed by tape casting and dry pressing.

the sinterability of these materials. A high homogeneous microstructure of the samples after firing can be observed in Fig. 8. At 1200 °C the firing shrinkages are equivalent between dry pressing and tape casting.

At 1220 °C the firing shrinkage for cast tape tiles is lower due to an expansion of the tiles caused by bubble formation during firing. The maximum shrinkage in tiles formed by tape casting takes place at a lower temperature; therefore, an expansion at higher temperatures is expected. Rounded pores can be observed in the microstructure of the material shown in Fig. 8.

3.2.3. Loss on ignition

Loss on ignition is the weight difference of the ceramic tile during the sintering process. Usually it is associated with the decomposition of organic matter and carbonates present in the raw materials, and evaporation of constitution water of minerals and of additives present in the mixture. A larger loss on ignition means higher volumes of gases released during firing. If the gases are fully released before the formation of vitreous phase during firing, defects like holes in the finished product are avoided [27]. In Fig. 4 a higher loss on ignition is observed for tiles formed by tape casting, due to the volatilization of the plasticizer (glycerol), which has a boiling point of 290 °C.

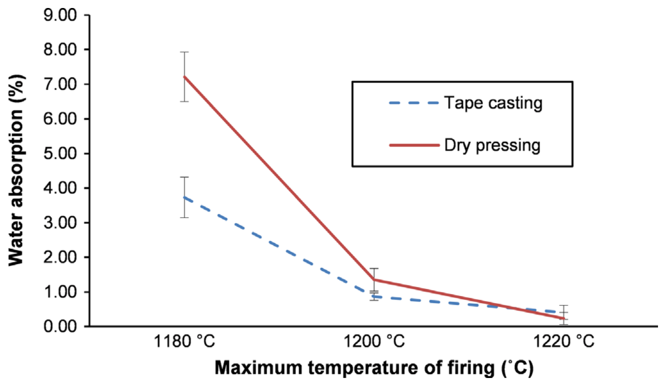

Fig. 7. Water absorption of the porcelain tiles formed by tape casting and dry pressing.

3.2.4. Mechanical strength

The mechanical strength measured as modulus of rupture presented higher values in the tiles formed by tape casting. With the maximum temperature of 1180 °C, the cast tiles showed mechanical strength 78% higher than those formed by pressing. At 1200 °C, this difference in mechanical strength was 86%. Fig. 5 shows the results of mechanical strength of the tiles investigated.

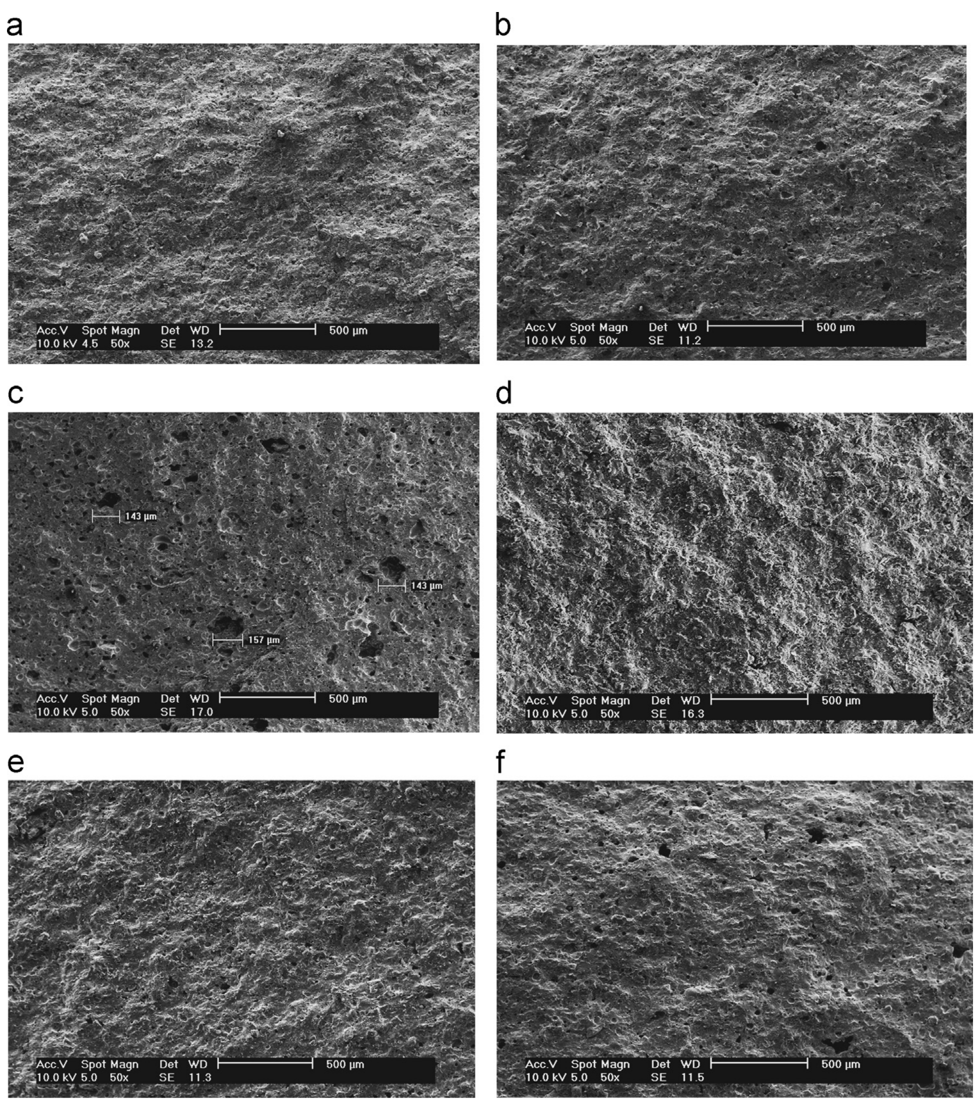

Fig. 8. Microstructure of the porcelain tiles formed by Tape Casting and Dry pressing. (a) Tape Casting 1180 °C, (b) Tape Casting 1200 °C, (c) Tape Casting 1220 °C, (d) Dry pressing 1180 °C, (e) Dry pressing 1200 °C and (f) Dry pressing 1220 °C.

The mechanical strength measured as breaking load presented higher values in the tiles formed by tape casting (Fig. 6). The ISO 13006 standard requires for tiles with water absorption minor than 0.5%, breaking load values above 700 N, so in both cases the breaking load is below the minimum required. Tiles with breaking strength less than 400 N are intended for use on walls only and this must be indicated by the manufacturer. Nevertheless, even when applying the product only in the wall, the breaking load should not be less than 200 N [28]. Thus, ceramic tiles with reduced thickness, as 2 mm, can be manufactured by tape casting for use on walls. The results show that at 1200 °C the breaking load for cast tape tiles is two times higher than ceramic tiles produced by dry pressing.

Taking into account that the characteristics of the process, such as chemical composition, particle size distribution, bulk density, maximum firing temperature, were the same for the two forming processes, it can be stated that the difference in mechanical strength was due to the forming process. Those results attest that tape casting is an alternative for production of thin porcelain tiles, providing even higher mechanical strength than those shaped by dry pressing.

The higher mechanical performance for tape cast samples is due to a more uniform microstructure, with less internal defects such as micro-cracks. This homogeneity is a function of a best arrangement of the particles during the liquid forming process.

3.2.5. Water absorption

The average of the water absorption between the two shaping methods and three firing temperatures are shown in Fig. 7. Lower water absorption for the tiles shaped by tape casting at 1180 °C is observed, comparing to the dry pressed samples. By tape casting is possible to achieve structures with least open pores at lower temperatures. At 1200 °C, water absorption between the methods presented minor differences, 0.87% for tape casting and 1.35% for pressing.

The temperature of maximum densification in the cast tiles is lower than the pressed tiles, what is in agreement to the high difference in water absorption between the methods at 1180 °C. The slight increase in water absorption in the tiles formed by tape casting and sintered at 1220 °C is another indication that the maximum densification of these tiles occurred at a lower temperature. At this latter temperature the expansion is higher, which can be evidenced by an amount of larger pores in their microstructure as shown in Fig. 8.

Those differences in water absorption between the two forming methods also show that it is possible to produce thin porcelain tiles by tape casting with lower water absorption processed at lower temperature, resulting in lower fuel consumption in the kiln.

3.2.6. Microstructure

The microstructure of the porcelain tiles presents a few visual differences for samples fired at 1180 and 1200 °C. However tiles fired at 1220 °C and formed by tape casting presented a greater amount of large closed pores, which can be seen in Fig. 8.

According to the liquid sintering process, the glassy phase formed involves practically all the pores and giving rise to closed porosity of the product. Due to the high surface tension of the film of liquid phase which surrounds the pores, the gas is trapped inside the compact [29].

The gas entrapped within the pores hinders the progress of the laminar flow of the vitreous phase, and as this flow advances, the pressure of gas trapped within the pores increases, due to the decrease in pore volume, and densification becomes increasingly difficult. The temperature rise, followed by thermal cycling, also contributes to the increased internal pressure of the gases, at the same time reducing the surface tension of the glassy phase. In a point, the internal pressure of the gases trapped within the pores exceeds the surface tension value of the vitreous phase, causing the increase of the pore volume [29].

Analyzing the values of water absorption and firing shrinkage in two forming methods it can be deduced that sintering occurs faster with the tiles shaped by tape casting. This can be confirmed by the higher shrinkage and lower water absorption at 1180 °C.

As the maximum densification of the tiles formed by tape casting occurs at a lower temperature, it is expected that the swelling of the pores also happen at a lower temperature, which can be evidenced by the larger pores shown in Fig. 8.

4. Conclusions

Tape casting can be considered an alternative manufacturing technique for production of thin porcelain tiles. The results show that for cast tapes, the sintering reactions occur at lower temperatures.

For porcelain tiles processed under the same conditions, the cast tapes presented a higher firing shrinkage at 1180 °C. At 1220 °C the firing shrinkage is lower, due to the increase of the sizes of internal pores of the material.

Considering the mechanical strength, tape casting presented higher values than pressing process. Moreover, the water absorption is lower at tape cast porcelain tiles.

The microstructures of the tiles formed by tape casting presents lower porosity at 1180° and higher at 1220 °C. The higher porosity at the latter temperature is due to the higher swelling on these tiles, as the maximum densification was reached at lower temperature.

Acknowledgments

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