

INFLUENCE OF PRESSING POWDER CHARACTERISTICS AND FIRING TEMPERATURE ON THE POROUS MICROSTRUCTURE AND STAIN RESISTANCE OF PORCELAIN TILE

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ABSTRACT

The excellent technical characteristics of porcelain tile compared to other kinds of flooring have led to a considerable increase in the use of this ceramic material in recent years. One of the problems that most impairs porcelain tile quality arises when its surface is polished with a view to enhancing the product's aesthetic qualities. Although polishing yields smooth glossy surfaces, some sealed pores that lie scattered throughout the tile are exposed at the surface, deteriorating the final product's stain resistance.

In this study, the influence of the pressing powder characteristics and peak firing temperature has been determined on the porous microstructure and stain resistance of porcelain tile.

Industrial spray-dried powders of the type commonly employed in porcelain tile manufacture were used in the first part of the study to determine the variation of true porosity and pore morphology with peak firing temperature. In the second part, pressing powders with differing characteristics were employed to compare the porous microstructure of the final product. In both cases, the relationship of the resulting porous microstructure to the polished product's ability to withstand staining was established.

The results obtained in this study show that it is possible to improve the stain resistance of polished porcelain tile by selecting the most suitable pressing powder characteristics and peak firing temperature so as to produce a microstructure that is made up of the minimum number of sealed and isolated pores.

1. INTRODUCTION

Porcelain tile is a product with a coloured body, generally with little surface decoration, which is homogeneous and has very low water absorption (usually below 0.1%). The manufacturing process basically consists of the following stages: wet milling and spray drying of the resulting suspension, followed by pressing, drying and firing, which usually takes place in roller kilns [1].

The low porosity of the fired tile largely determines its excellent mechanical and surface properties. Thus, porcelain tile is generally characterized by its high mechanical strength, abrasion and frost resistance, as well as its unalterability when exposed to common chemical agents (acids, cleaning products, alkalis, etc.).

One of the factors that most impairs porcelain tile quality arises when the tile surface is polished to enhance its aesthetic characteristics. Although such treatment produces smooth and highly glossy surfaces, pores that were sealed within the body appear at the surface, thus lowering the stain resistance of the installed tile.

To reduce this defect, tile must be produced with minimum sealed porosity. This property is mainly determined by the characteristics of the composition, and the manufacturing process variables. In industrial practice, it has often been observed that when the composition contained an excessive proportion of impurities (Fe_2O_3 , carbon, etc.), or the processing had not been appropriate (insufficient degree of milling, inadequate pressing or firing, etc.), materials were obtained with low stain resistance. Therefore, to obtain low sealed porosity, besides minimising the presence of impurities that break down at high temperature, the formed tile must exhibit a suitable microstructure to facilitate the suppression of the pores present in the firing stage. On the other hand, it is vital that the liquid phase that arises during this stage should exhibit the appropriate viscosity to allow obtaining a high density product unaffected by defects relating to dimensional non-uniformity.

Thus, if the characteristics of the starting composition and/or values of the process variables are unsuitable, a product will be obtained with excessive sealed porosity and therefore with decreased stain resistance.

On the other hand, in view of the differing stain resistant behaviour of porcelain tile found in the marketplace, whose sealed porosity values are quite alike, it seems logical to assume that it is not only this property that determines stain resistance, but also all the remaining characteristics of the fired tile, which define its porous microstructure (pore-size distribution, pore shape and connectivity, etc.).

Although studies exist [2][3] on the influence of the forming process variables (moisture content and pressure) on some porcelain tile characteristics (porosity, firing shrinkage, mechanical strength, etc.), no rigorous study has been undertaken to date of the causes of sealed or final porosity. Nor have studies been encountered in the literature, which have attempted to relate the porous microstructure of the fired tile with the fired product's ability to withstand staining.

This study was therefore undertaken with a view to determining the relationship between the porous structure of porcelain tile and its stain resistance. For this purpose, using industrial compositions, the impact of pressing powder characteristics (presence or absence of agglomerates, agglomerate size, etc.), and peak firing temperature on tile porous

microstructure were determined, in an intent to relate this microstructure to the polished product's stain resistance.

The work was thus aimed at learning more about the most suitable operating conditions for porcelain tile manufacture with a view to obtaining porcelain tile with enhanced quality.

2. EXPERIMENTAL

2.1. MATERIALS

Two industrial spray-dried powders of the kind that are commonly employed in porcelain tile manufacture were used as starting materials (PI1 and PI2).

The first was used to study the effect of peak firing temperature on the porous microstructure of the tile. The second powder sample was employed to determine how the agglomerate characteristics influence this same property. Both spray-dried powders were formulated from natural kaolinitic-illitic clays, potassium-sodium feldspar and feldspathic sand. The main difference between the two spray-dried powders was the degree of milling that each had undergone.

The chemical composition of the spray-dried powders was determined by X-ray fluorescence. Particle-size and agglomerate-size distribution were respectively determined by X-ray absorption (with the Sedigraph technique) and dry screening. These results have been detailed in Tables I and II and in Fig. 1. Finally, the specific surface area, assessed by the BET method, was 15.2 and 14.8 m²/g respectively for samples PI1 and PI2.

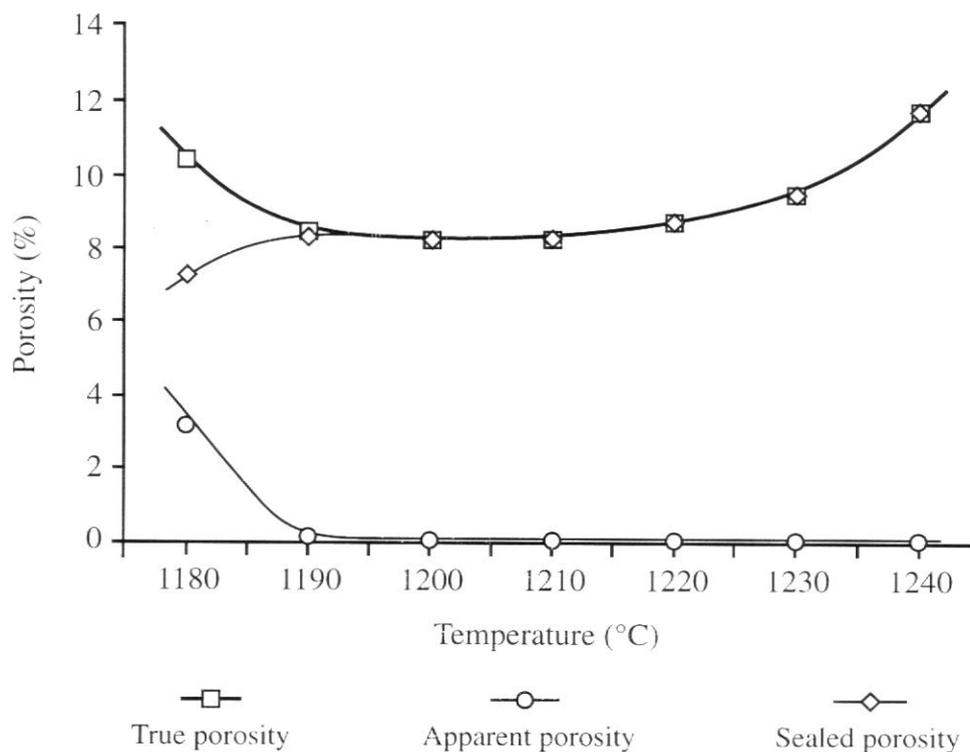


Figure 1. Particle-size distribution of spray-dried powders PI1 and PI2.

Table I. Chemical composition of the industrial spray-dried powders (wt%).

SiO ₂	67.70
Al ₂ O ₃	20.10
Fe ₂ O ₃	0.62
CaO	0.63
MgO	0.23
Na ₂ O	4.28
K ₂ O	1.41
TiO ₂	0.87
Loss on ignition	4.05

Table II. Agglomerate-size distribution of the industrial spray-dried powders (wt%).

Mesh aperture (μm)	Differential percentage (%)
> 600	4
500-600	13
300-500	56
200-300	20
< 200	7

2.2 EXPERIMENTAL PROCEDURE

2.2.1 Pressing powder preparation

Two pressing powders were prepared from industrial spray-dried powder PI2, referenced PF and PD. The first of these (PF) corresponded to a particle-size fraction of sample PI2 in the range 100 to 200 μm, and was obtained by dry screening. The second (PD) was prepared by breaking up the agglomerates present in the industrial spray-dried powder in an agate mortar.

2.2.2 Test specimen forming

The four prepared pressing powders (PI1, PI2, PF and PD), were used to form 0.7 cm thick cylindrical test specimens, with a diameter of 4 cm, by unidirectional pressing in a laboratory hydraulic press. Pressing moisture content for all the powders was 0.055 kg water/kg dry solid. Test specimens were formed from the industrial spray-dried powder PI2 at pressing pressures of 400 and 800 kg/cm² (PI400 and PI800). The remaining test specimens from the other spray-dried powders were all formed at 400 kg/cm².

The test specimens obtained were dried at a temperature of 110 °C in an electric laboratory oven with recirculating air to constant weight (determined on scales with an accuracy of 0.001g). They were subsequently measured using callipers with an accuracy of 0.01mm, and bulk density was determined by the method involving immersion in mercury [4].

2.2.3 Test specimen firing

The dry test specimens were fired in an electric laboratory kiln at different peak firing temperatures, ranging from 1180 °C to 1240 °C. The heating rate was 25 °C/min, with a 6 min dwell at peak temperature. Cooling took place by forced convection, using a ventilation system, except at temperatures in the range 590 to 550°C, at which natural convection was employed.

2.2.4 Characterization of the fired test specimens

Bulk density of the test specimens (ρ_C) was determined by immersion in mercury. Linear firing shrinkage was assessed as the difference between the diameter of the dry and fired specimen, defining this parameter on a dry basis. Water absorption (E) was calculated by measuring the weight gain of the test specimens on placing them in boiling water for two hours, according to the procedure set out in standard EN 99.

These values allowed calculating true porosity (ϵ_T), apparent porosity (ϵ_a) and sealed porosity (ϵ_C) of the test specimens by means of the following equations:

$$\epsilon_T = \left(1 - \frac{\rho_C}{\rho_r}\right)_{100} \quad (1)$$

$$\epsilon_a = E \cdot \rho_C \quad (2)$$

$$\epsilon_C = \epsilon_T - \epsilon_a \quad (3)$$

where ρ_r is the true density of the fired product, determined on specimens fired at 1200°C with a helium pycnometer, making the assumption that this was then constant for the remaining temperatures. The value found was 2.60 g/cm³.

Finally, in order to examine the porous microstructure and assess the stain resistance of the materials, cross-sectional slices were prepared from the centre of the test specimens. These specimens were then embedded in an epoxy resin and polished, upon which they were stained with a special felt-tipped pen used for glass. After one minute, the samples were vigorously cleaned using cotton wool soaked in alcohol, and subsequently examined by optical microscopy using two illumination systems: bright- and dark-field illumination. In the first, direct lighting was involved, at right angles to the inspected surface, allowing the pores and surface irregularities (microstructure) to be observed, whereas in the dark-field, the lighting was indirect, yielding images that are similar to those perceived visually by the human eye. The latter technique allows perceiving and quantifying dirt retention in the pores.

3. RESULTS

Table III details the results corresponding to the characterization of the unfired and fired test specimens formed from the four types of studied powders (PI1, PI2, PF and PD), as well as those of the series of test specimens formed using powder PI2 at greater pressure (PI800).

Table III. Characteristics of the unfired and fired test specimens formed from the four studied pressing powders.

Ref.	Dry bulk density (g/cm ³)	Temperature (°C)	Linear firing shrinkage (%)	Water absorption (%)	Fired bulk density (g/cm ³)	True porosity (%)	Apparent porosity (%)	Sealed porosity (%)
PI1	1.850	1180	8.1	1.34	2.330	10.38	3.12	7.26
		1190	8.7	0.04	2.381	8.43	0.10	8.33
		1200	8.7	0.01	2.386	8.22	0.01	8.21
		1210	8.7	0.01	2.386	8.22	0.02	8.20
		1220	8.6	0.05	2.374	8.68	0.02	8.66
		1230	8.4	0.02	2.354	9.45	0.05	9.40
		1240	7.7	0.01	2.295	11.72	0.03	11.69
PI400	1.877	1180	7.9	1.43	2.321	10.73	3.32	7.41
		1190	8.5	0.06	2.372	8.77	0.14	8.63
		1200	8.5	0.00	2.377	8.58	0.00	8.58
		1210	8.5	0.02	2.377	8.58	0.05	8.53
		1220	8.4	0.00	2.365	9.04	0.00	9.04
		1230	8.2	0.02	2.345	9.81	0.05	9.76
		1240	7.5	0.03	2.286	12.08	0.07	12.01
PI800	1.979	1180	6.8	0.13	2.373	8.73	0.31	8.42
		1190	7.0	0.03	2.389	8.12	0.07	8.04
		1200	7.0	0.02	2.391	8.04	0.05	7.99
		1210	6.8	0.01	2.375	8.65	0.02	8.63
		1220	6.6	0.02	2.363	9.12	0.05	9.07
		1230	6.2	0.01	2.330	10.38	0.02	10.36
		1240	5.3	0.02	2.249	13.50	0.04	13.46
PD	1.961	1180	7.1	0.11	2.387	8.19	0.26	7.93
		1190	7.2	0.04	2.401	7.65	0.10	7.56
		1200	7.3	0.04	2.408	7.38	0.10	7.29
		1210	7.2	0.02	2.396	7.85	0.05	7.80
		1220	7.0	0.01	2.391	8.04	0.02	8.01
		1230	6.7	0.02	2.362	9.15	0.05	9.11
		1240	6.3	0.02	2.325	10.58	0.05	10.53
PF	1.875	1180	7.9	1.01	2.319	10.81	2.34	8.47
		1190	8.4	0.11	2.359	9.27	0.26	9.01
		1210	8.5	0.04	2.374	8.69	0.09	8.60
		1230	8.1	0.04	2.339	10.04	0.09	9.94
		1240	7.5	0.00	2.279	12.35	0.00	12.35

3.1 INFLUENCE OF FIRING TEMPERATURE ON PORCELAIN TILE POROSITY.

Fig. 2 depicts a plot of the evolution of true porosity, apparent porosity, and sealed porosity, of the test specimens formed at 400 kg/cm² from powder PI1 as a function of peak firing temperature. It can be observed that as peak firing temperature rises, true porosity falls as a result of the sintering process that arises in the presence of liquid phase.

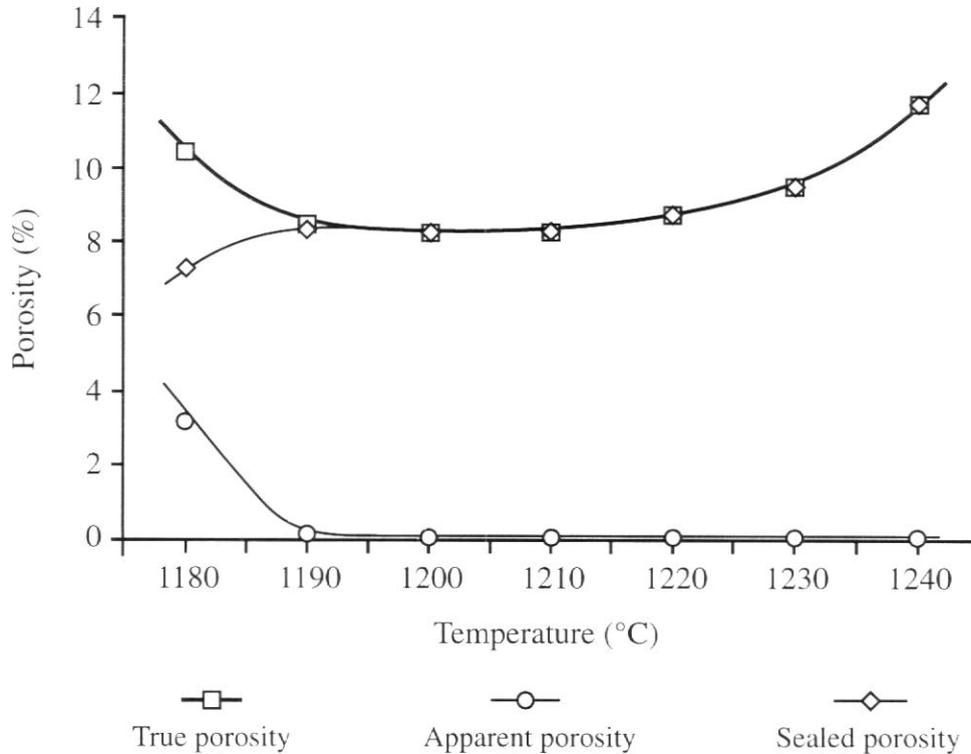


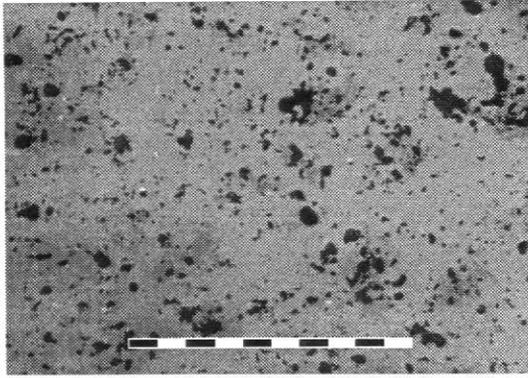
Figure 2. Variation of true, apparent and sealed porosity of the test specimens formed from powder PI1 with peak firing temperature.

The figure shows that densification of the material is mainly due to the decrease in its apparent porosity, which becomes virtually zero at 1200 °C. This drop in apparent porosity is associated with a rise in the specimen's sealed porosity, as a result of the progressive incommunication of the pores that takes place on increasing the glassy-phase proportion [2][5].

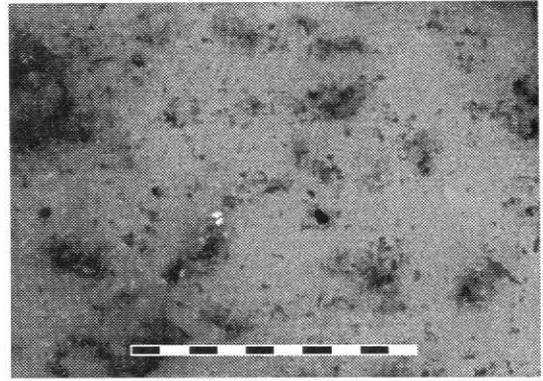
At temperatures beyond 1200°C (at which almost all porosity is sealed porosity), the pressure of the occluded gases within the pores rises, opposing the progressive densification of the material. Sealed porosity thus grows with temperature, even after the virtual suppression of apparent porosity, yielding a volume increase or bloating of the tile.

3.2 RELATIONSHIP BETWEEN THE POROUS MICROSTRUCTURE AND STAIN RESISTANCE OF PORCELAIN TILE

In order to relate the microstructure of the test specimens to their stain resistance, polished cross-sectional slices of the test specimens, which had been formed at 400 kg/cm² from industrial spray-dried powder PI1, were examined by optical microscopy after the stain resistance tests were carried out. These specimens had been fired at the following peak firing temperatures: 1180, 1200, 1210 and 1220°C. Figs. 3 to 6 show the photographs taken under bright-field and dark-field illumination. It should be kept in mind that in the bright-field images all the pores present at the specimen surface can be observed, whereas in the dark-field images, only those can be perceived that have retained dirt.

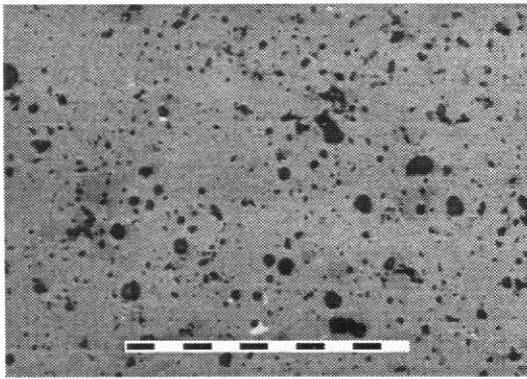


Bright-field

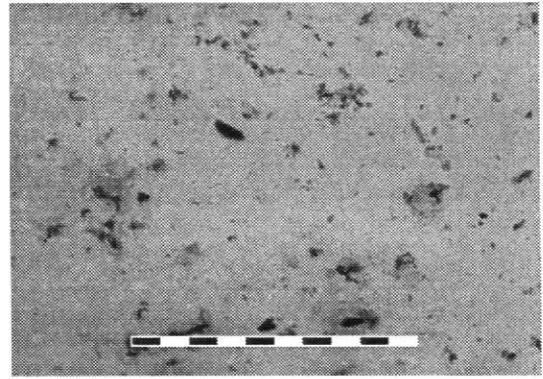


Dark-field

Figure 3. Photographs corresponding to the PI1 series test specimen fired at 1180°C. (1000 μm)

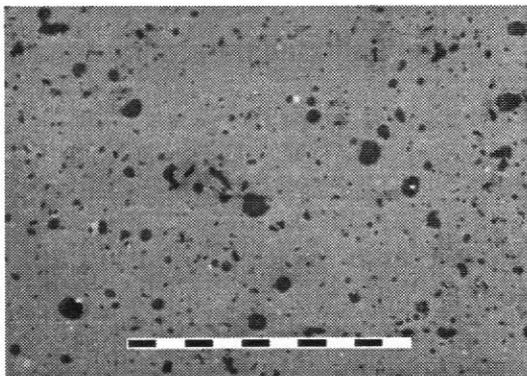


Bright-field

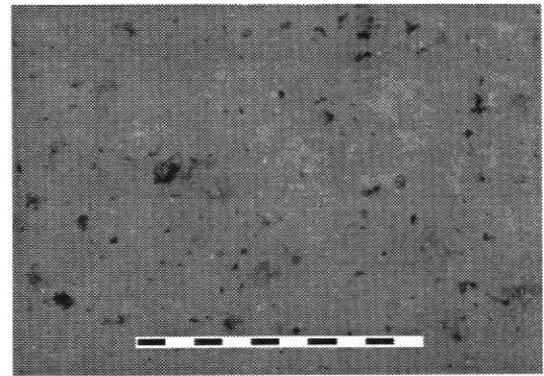


Dark-field

Figure 4. Photographs corresponding to the PI1 series test specimen fired at 1200°C. (1000 μm)

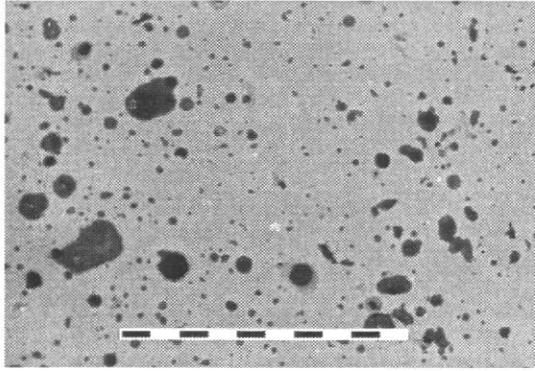


Bright-field

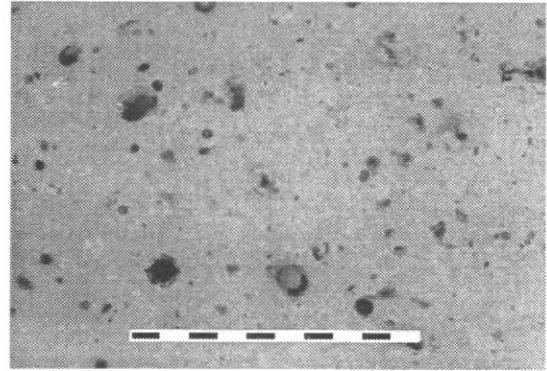


Dark-field

Figure 5. Photographs corresponding to the PI1 series test specimen fired at 1210°C. (1000 μm)



Bright-field



Dark-field

Figure 6. Photographs corresponding to the P11 series test specimen fired at 1220°C. (1000 μm)

The figures show that as firing temperature rises, dirt retention decreases, up to a temperature (1210°C), beyond which this trend is inverted. This behaviour seems to be related to the variation that the porous microstructure of the tile undergoes with firing temperature.

Thus, at low temperatures, below peak densification temperature (Fig. 3), the porous microstructure of the material is made up of a system of interconnected pores, into whose numerous capillaries dirt enters, which can subsequently not be removed, making the tile surface look stained.

On raising temperature, the porous texture evolves towards a system with fewer interconnected pores, reducing the number of small pores (below 5 μm) and slightly raising the volume of the larger-sized ones (above 30 μm). Consequently, surface quality as far as a stained appearance is concerned, improves noticeably, as Fig. 4 shows.

Finally, at temperatures above 1200°C, at which the material bloats, the porosity observed is mainly comprised of isolated, round pores, as a result of the pressure exerted by the gases trapped inside the pores. At these temperatures, if the degree of bloating does not become excessive (1210°C), the surface appearance of the specimen continues to improve. However, if advanced bloating occurs (1220°C), surface quality starts deteriorating, owing to the presence of large pores (exceeding 60 μm), which retain dirt.

It is worth pointing out that when the porous microstructure is principally made up of isolated pores, their cleanability does not depend upon the size of pore exposed at the surface, rather on its depth. Thus, large pores may exhibit greater cleanability than smaller ones, as Fig. 6 shows. However, from a statistical point of view, the depth that the pores at the polished surface exhibit is related to their size, allowing the inference that the most suitable firing temperature for this type of product, with regard to its behaviour when exposed to staining agents, must be that which provides the tile with a microstructure consisting of a minimum number of small, isolated pores (below 15 μm). Such conditions are found in standard porcelain tile compositions at firing temperatures slightly exceeding peak densification temperature.

3.3 INFLUENCE OF PRESSING POWDER CHARACTERISTICS ON THE POROUS MICROSTRUCTURE AND STAIN RESISTANCE OF PORCELAIN TILE.

Fig. 7 shows a plot of the variation of true porosity of the specimens formed from the studied spray-dried powders (PI2, PF and PD) with peak firing temperature. It can be observed that the variation of true porosity with temperature for samples PF and PD is qualitatively quite similar to that found for a spray-dried powder, whose behaviour was described in Section 3.1.

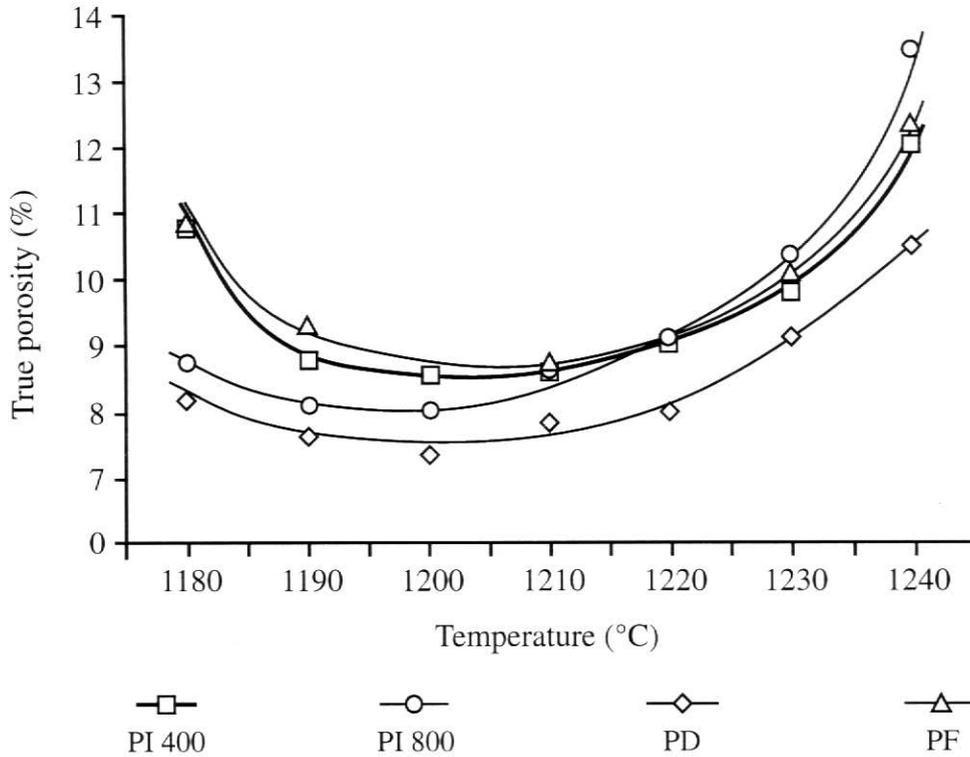


Figure 7. Variation of true porosity with peak firing temperature of the test specimens corresponding to series PI400, PI800, PD and PF.

A comparison of the curves corresponding to the tests specimens formed at 400 kg/cm² from spray-dried powder PI2 (PI400) and fraction 100-200 μm of that powder (PF), shows no significant differences in the value of the minimum porosity obtained. This result suggests that for the tested spray-dried powder, the size of the agglomerates has no significant effect upon the resulting final porosity.

On comparing the curves corresponding to the industrial spray-dried powder (PI400) and the disagglomerated powder obtained from it (PD), it can be observed that the specimens formed from the latter material exhibited less porosity across the whole range of tested firing temperatures, yielding fired materials with lower final porosity. Such differing behaviour might be related to the presence of microstructural heterogeneities in the pressed specimens formed from the spray-dried powder, which could in turn come from the granule's own microstructure and/or the presence of agglomerates or agglomerate fractions that were not broken up or deformed. According to the literature surveyed [6-9], agglomerates obtained by spray-drying are characterized by a lack of microstructural uniformity, mainly owing to the void or intragranular pore, as well as the marked orientation of the plate-like particles of clay mineral within the granule.

However, as a drop has been reported in some studies in final tile porosity with the increase in compactness of the unfired body [2], the differences that have been observed could be related to this fact, since the test specimens formed from the disagglomerated powder (PD) showed greater compactness of the unfired body (Table III) in respect of the test specimens formed from the industrial spray-dried powder (PI400).

To compare this last aspect, test specimens were formed from spray-dried powder PI2 at greater pressure (PI800) and fired at different temperatures. The variation of porosity with peak firing temperature for these specimens has also been plotted in Fig. 7. The figure shows that in spite of the fact that this last series of test specimens (PI800) exhibited greater dry bulk density than the test specimens formed from the disagglomerated powder, lower final porosity was not found in the fired materials. However, final porosity was lower than that obtained from the series of test specimens formed at lower pressure (PI400), as expected in view of the data reported in foregoing studies.

These results show that although the granules present in the spray-dried powder must be virtually broken up or deformed at the pressures usually employed for forming such tile ($350\text{-}600\text{ kg/cm}^2$), the microstructure that is produced when a spray-dried powder agglomerate is used appears to be the cause of a certain final porosity in the fired materials. This effect is not observed when the powder involved contains no agglomerates.

With a view to verifying whether the differences in final porosity that had been observed yielded differing behaviour with regard to staining agents, the stain test was carried out (Section 2.2.4) on the test specimens fired at a temperature of 1210°C . The photographs in Figs. 8 to 11 show the results. It can be observed in these photographs that the test specimens formed at higher pressure (PI800) and to a larger extent the specimens corresponding to the disagglomerated powder (PD), exhibit a better surface appearance (less staining) than the series formed at 400 kg/cm^2 (PI400). The behaviour observed is not only due to their low final porosity but the smaller number of pores capable of retaining dirt, which are basically medium-sized ($30\text{ }\mu\text{m}$) or large pores (exceeding $60\text{ }\mu\text{m}$).

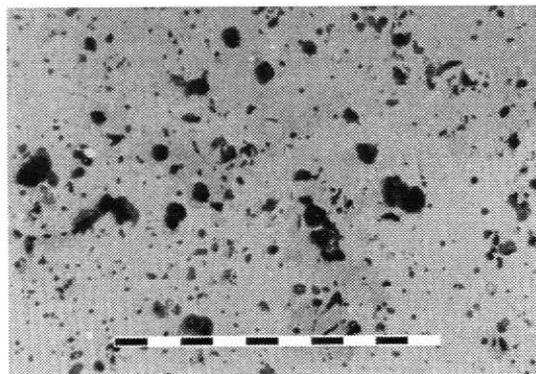
Similar results to the foregoing have been found in test specimens formed from powder PF. It can be observed on comparing the photographs in Figs. 8 to 11, corresponding to the test specimens formed from industrial powder (PI400) and particle-size fraction $100\text{-}200\text{ }\mu\text{m}$ of this powder (PF), that the presence of fewer large pores in these specimens, probably as a result of the existence of smaller intragranular pores or voids, noticeably improves the surface quality of the materials, thus lowering the impression of staining.

3.4 ASSESSMENT OF THE CAUSES GIVING RISE TO FINAL POROSITY IN PORCELAIN TILE FORMED FROM SPRAY-DRIED POWDER AGGLOMERATES.

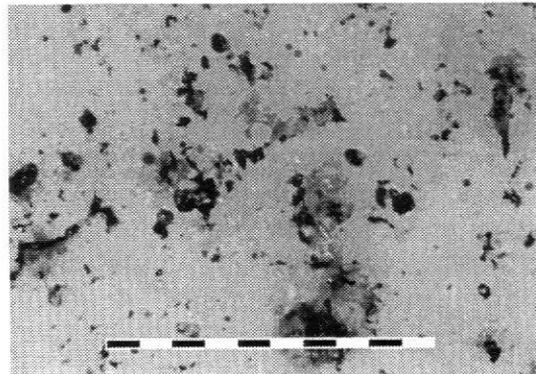
In the previous sections, it has been shown that when a spray-dried powder is used, materials with a certain final porosity are obtained, associated to the very characteristics of the agglomerates making up the powder sample. This porosity, mainly consisting of medium-sized or large pores (exceeding $30\text{ }\mu\text{m}$), may sometimes (depending upon the compositional characteristics and the values of the process variables) appreciably lower the product's ability to withstand staining.

With a view to substantiating the above results, while simultaneously trying to determine the inter- or intragranular nature of final porosity, polished cross sections of test specimens fired at different peak firing temperatures were examined by optical microscopy. These test specimens had been formed from a pressing powder made up of a

mix of three parts of a coloured spray-dried powder (obtained by adding 2 wt% black pigment) to one uncoloured part. Thus, on superimposing the images obtained in the bright-field and the dark-field by means of an image analyzer connected to the microscope, the pores existing at the polished specimen surface and the outlines of the agglomerates could be simultaneously visualized. The procedures used for forming, firing, and polishing the test specimens were identical to the ones described before. Although a considerable number of regions were inspected in each test specimen, the images corresponding to only one representative region of each test specimen are shown in this study.

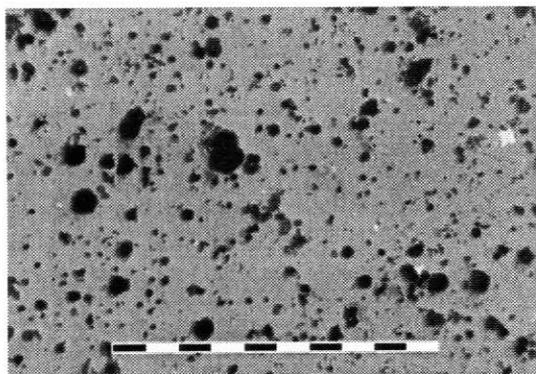


Bright-field

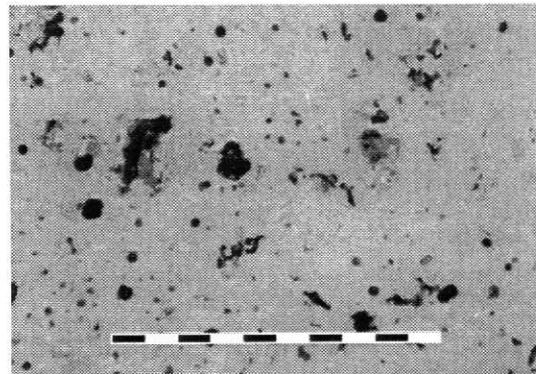


Dark-field

Figure 8. Photographs corresponding to the PI400 series test specimen fired at 1210°C. (1000 μm)

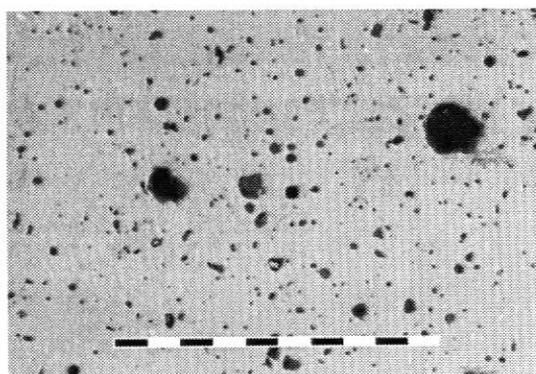


Bright-field

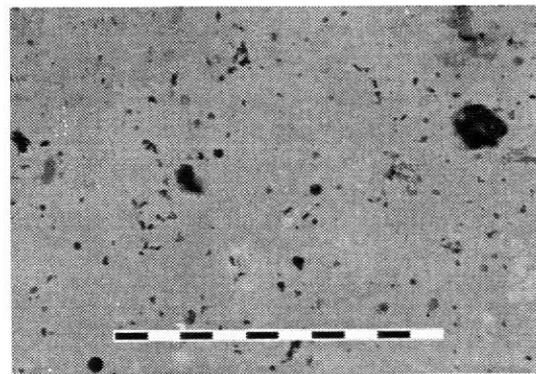


Dark-field

Figure 9. Photographs corresponding to the PI800 series test specimen fired at 1210°C. (1000 μm)



Bright-field



Dark-field

Figure 10. Photographs corresponding to the PD series test specimen fired at 1210°C. (1000μm)

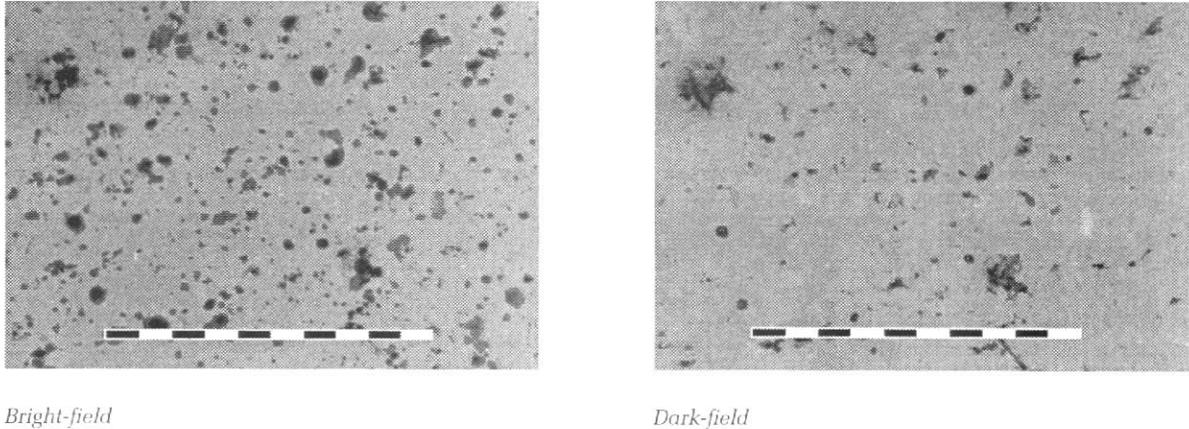


Figure 11. Photographs corresponding to the PF series test specimen fired at 1210°C. (1000 μm)

Figs. 12 to 15 depict the images obtained. The existing pores are shown as black, the agglomerates corresponding to the spray-dried powder with pigment are a dark grey, and the ones from the powder without pigment are a light grey colour. It can be observed that at temperatures below peak densification temperature (1180°C), a part of the existing pores, having large sizes (60-120 μm), appears to be linked to the presence of agglomerates that have not been fully broken up or deformed during pressing, and have kept their identity in the fired material. Thus, Fig. 12 clearly shows the outlines of the agglomerates just as the presence of large pores in the central region of the agglomerates, which may be related to the characteristic inner void of agglomerates produced by spray drying.

On raising temperature up to values close to peak densification temperature (1200°C), it can be observed that as a result of the sintering process, the smaller pores progressively disappear, whereas the larger ones remain and even grow, some of which are located in the central region of the agglomerate.

At higher temperatures (above 1200°C), at which the material starts bloating, some pores may still be identified together with agglomerates, however as peak firing temperature rises, agglomerate outlines become more diffuse as a result of the advanced state of sintering, which hinders establishing a clear relationship between the presence of large pores and the existence of partially broken up or deformed agglomerates.

These results suggest that at least a part of the medium-size or large pores are related to the use of spray-dried powders, and more specifically to the characteristic intragranular pore or void of the agglomerates that comprise this pressing powder. However, the results obtained do not allow identifying with certainty at which moment these pores arise, since they may be due to the presence of agglomerates that were not wholly broken up or deformed in pressing, or to their reappearance in firing at the position they originally took up. This latter effect could arise as a result of the behaviour during sintering of the materials formed from agglomerates characterized by a marked microstructural heterogeneity. An example of such behaviour could be the anisotropic shrinking that this kind of agglomerate undergoes as a result of the orientation exhibited by the clay mineral particles that they contain [10].

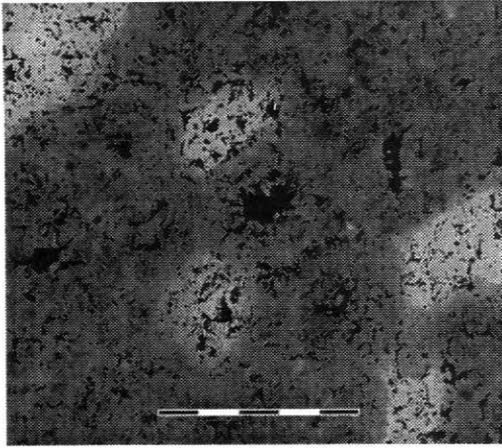


Figure 12. Photograph of the test specimen fired at 1180°C, formed from a spray-dried powder mix with and without pigment. (500µm)

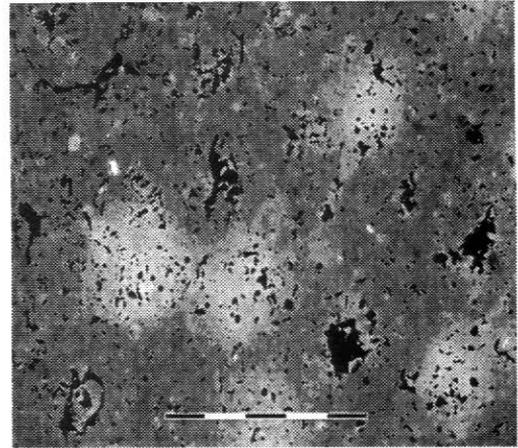


Figure 13. Photograph of the test specimen fired, at 1200°C shaped from a spray-dried powder mix with and without pigment. (500 µm)

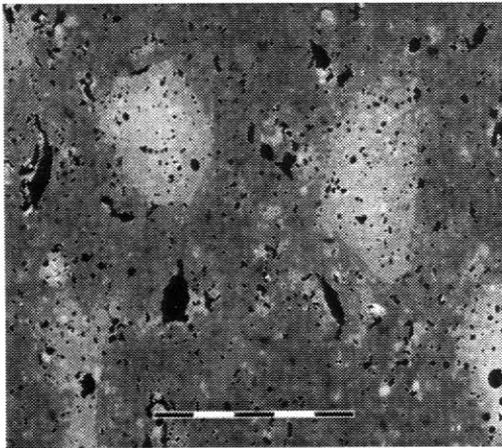


Figure 14. Photograph of the test specimen fired at 1220°C, formed from a spray-dried powder mix with and without pigment. (500µm)

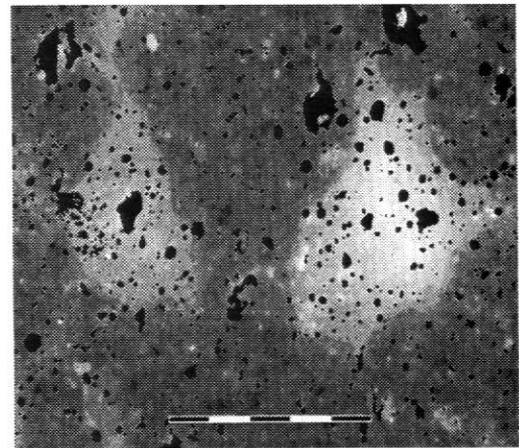


Figure 15. Photograph of the test specimen fired at 1240°C, formed from a spray-dried powder mix with and without pigment. (500µm)

4. CONCLUSIONS

The results obtained in this study allow drawing the following conclusions:

The porous microstructure that provides polished porcelain tile with suitable stain resistance is made up of the minimum number of small, isolated pores (smaller than 15 microns).

For a given porcelain tile composition, the most suitable firing temperature for obtaining the targeted porous microstructure lies at slightly higher values than those of the temperature at which peak densification of the material occurs.

On working under the foregoing conditions, raising pressing pressure yields porcelain tile with greater stain resistance, as a result of the decrease in final porosity and the number of medium-size or large pores (above 30 microns). On using pressing powder made up of small-sized agglomerates (100-200 microns), although the resulting fired materials

have practically the same porosity as the ones obtained from industrial spray-dried powder, their stain resistance is greater, possibly because the smaller agglomerates also contain smaller voids.

A procedure has been designed, involving the use of an optical microscope hooked up to an image analyzer, which allows distinguishing the inter- or intragranular nature of final porosity in porcelain tile. Using this technique, it has been shown that an important proportion of large pores (exceeding 60 microns), which make up this porosity is related to the use of spray-dried powders.

It has been shown that using a non-agglomerate pressing powder allows producing materials with greater density and stain resistance than when a spray-dried powder is used. The micro-heterogeneities that are present (agglomerates with a non-uniform microstructure, intragranular porosity, etc.), seem to affect the sintering process, giving rise to a porous microstructure consisting of a bigger proportion of large pores.

The results obtained in the present study can lead to new lines of research aimed at developing new pressing powders that will yield porcelain tile with enhanced quality.

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