Chapter 2
Porcelain Tile

Abstract This chapter described all features and characteristics of the porcelain tile such as the raw materials and the ceramic processing to produce the porcelain as well as some properties defined by the norms. Some aspects of pressing, drying and fire are also presented.

Keywords Porcelain tile · Ceramic processing · Norms

Porcelain stoneware tile is without doubt the class of ceramic coating which most evolved in recent decades, both in terms of technical properties as in relation to marketing. According to Heck (1996), porcelain stoneware tile emerged in the 1980s, being basically used in industrial and commercial flooring with very few color options and manufactured in small formats. The author mentions two important changes in the porcelain industry that allowed this evolution. The first was the replacement traditional tunnel ovens by modern roller ovens, allowing the manufacture of products with large formats, the most significant change was the transformation of porcelain stoneware tile in a modern and versatile material through the polishing process and the introduction of decorating techniques.

Porcelain tile is certainly a product of higher quality compared to traditional stoneware, resulting from the use of carefully selected raw materials and processing procedures accurately controlled. The emergence of the porcelain tile is partly associated with a conceptual and technological development in all the phases of the production process, from the reformulation of masses to the redefinition of the firing cycles, and also to the development of coloring and decorating techniques (Oliveira 1998).

The porcelain designation is sufficient to define the origins and characteristics of the product. Indeed, in the ceramic terminology stoneware indicates a very compact material composed of various crystalline phases dispersed in a glassy matrix, while the adjective porcelain has an etymological root in the term porcelain, the noblest ceramic material in evidence and appreciated for centuries (Oliveira 1998). According to Biffi (2002), porcelain tile is the product that offers the highest
possible interventions on the production lines and with which ceramic technicians
have acquired the pleasure of “making pottery” because they can invest in this
product all the expertise they have. Therefore, large sums are invested in research
for product innovation and process innovation for ceramic plates of porcelain tiles.
According to Heck (1996), porcelain tiles stand out due to the following features:

- High abrasion strength;
- Resistance to frost;
- Resistance to acids and alkalis;
- Uniformity of colors;
- Waterproofing;
- Easy maintenance;
- Various composition possibilities.

It is noteworthy that the excellent technical properties of porcelain tiles are
achieved through careful control of the entire ceramic processing, which starts in
the careful selection of raw materials and final inspection for packaging and ship-
ping of the product. All that is required to obtain the desired microstructure, which
should be as compact as possible, with minimum porosity and exclusively closed,
i.e., any existing pores must be closed without interconnection with the outside
of the ceramic body. Thus, with the desired microstructure, the desired properties
and quality of the manufactured product are maximized. The accurate control of
ceramic processing with a firing temperature ranging from 1,200 to 1,250 °C, are
the factors responsible for the high added value of porcelain tiles.

In Brazil, ceramic coatings are classified according to NBR 13817 using solely
water absorption (WA) as a parameter for the classification of ceramic coating as
described in Table 2.1.

According to NBR 13817, porcelain tile belongs to the ceramic group Bla, with
AA less than or equal to 0.5 %.

WA is a property that measures the amount of water that the ceramic body is
able to absorb within a normalized period of time. To be determined through an
extremely practical test and also reveal a notion of the ceramic body behavior
in relation to porosity, WA was for a long time, the main parameter to classify
a ceramic plate. Intuitively, the lower the WA, the more compact and dense the
material microstructure; this was the way the porcelain quality was assessed
until the NBR 15463 entered into force in February 2007. This standard is spe-
cific for porcelain tiles and determines the minimum values of the main proper-
ties that the product should achieve. According to ANFACER, NBR 15463 is the

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<th>Water absorption (%)</th>
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<td>Al</td>
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<td>0.5 &lt; WA &lt; 3</td>
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<td>WA &lt; 10</td>
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Table 2.1   Classification of ceramic tiles according to NBR 13817
first specific standard for porcelain tiles in the world and aims to raise the quality of products and protect consumers from misuse of the term “porcelain tile” about products whose characteristics do not comply with the new Brazilian standard. This standard defines “porcelain tile” as: “Ceramic plates for coating consisting of clays, feldspars, and other inorganic raw materials. It is intended to coat floors and walls, and can be shaped by pressing, extrusion, or other processes. The manufacturing process involves a high degree of grinding, high content of fluxing raw materials and high densification upon firing, resulting in products with low porosity and high technical performance. It can be glazed or not, polished or natural, rectified or not. Porcelain tile may have various dimensions: Small (product area < 50 cm²), intermediate (50 cm² < Product area < 2,500 cm²) or large size (product area > 2,500 cm²”).

NBR (Brazilian Norm) 15463 divided porcelain tiles into two categories: technical porcelain tile and glazed porcelain tile and also added other technical properties such as “performance evaluators,” apart from water absorption (wa). Table 2.2 shows the main properties and characteristics of porcelain tiles according to NBR 15463.

Therefore, NBR 15463 undoubtedly provides the classification of porcelain tiles using physical properties (water absorption, flexural strength, abrasion resistance, and tensile strength) and chemical properties and parameters (resistance to staining and to chemicals) and geometry of plates (side straightness, orthogonally, central and lateral bending, and twist).

### 2.1 Ceramic Processing of Porcelain Tiles

#### 2.1.1 Raw Materials

Ceramic coverings are manufactured by ceramic processing from the dust, where a mixture of raw materials, known by ceramists as “ceramic mass,” undergoes a sintering process (Petzold 1992). A ceramic mass for porcelain tile production
Porcelain Tile consists of raw material of distinctive characteristics, which can be classified into three main groups: (a) plastic, (b) fluxing agents and (c) inert materials, with compositions ranging within the region shown in the ternary diagram of Fig. 2.1.

The plastic materials used are clay and kaolin, which main function is to provide plasticity to the mass and therefore green resistance to the pressed body. The main flux materials used are feldspar, talc and pyrophyllite, whose main role is to produce liquid phase during sintering. As inert raw materials, quartz is undoubtedly the most common due to its immense availability, and as inert materials, they essentially remain inert during the firing cycle and are used mainly for the purpose of improving the dimensional stability after firing. Detailed information and features of each raw material are described below.

Every ceramist knows that a classical whiteware must have clay (50 %), feldspar (25 %), and flint (25 %). The main function of the clay is to provide plasticity and workability to form different suitable shapes. The feldspar acts as a flux during firing and the flint, usually quartz is inert filler, providing strength to the dried and fired specimens (Brownell 1976; Acchar 2006).

### 2.1.2 Clay

According to Santos (1989), clay is a natural, earthy and fine-grained material, which generally acquires certain plasticity when moistened with water; chemically, clays are composed of hydrated silicates of aluminum, iron and magnesium, containing certain amounts of alkali and alkaline earth elements. There is considerable variation in the terminology of clay minerals in various scientific and technological fields that use this material. As described above, plasticity is the main reason for the use of clays for ceramic masses, and according to Santos (1989), plasticity in clays is mainly a result of attraction forces between clay particles and the lubricant action of water between anisometric lamellar particles. It could be assumed

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**Fig. 2.1** Ternary diagram highlighting the region of compositions of a ceramic mass

![Ternary diagram](image_url)
that plasticity develops when clay has enough water to cover the entire accessible surface of clay minerals with a film that acts as a lubricant facilitating the sliding of the plates on each other when a shear stress is applied. Como-oriented water molecules are trapped on the surface of the clay minerals through hydrogen bonds, they also serve to bind clay particles to each other in the wet clay form, giving rise to various forms of mechanical strength of the green clay.

Biffi (2002) states that in addition to plasticity, clays used in ceramic masses for porcelain tiles need to present additional features as below:

- To provide light coloring during firing;
- To provide rheological properties facilitating flow;
- To provide with the flux characteristics a good density in firing;
- To provide excellent mechanical characteristics in firing.

To meet the characteristics described above, especially in relation to the light coloring during firing, kaolinite clays are preferably used, evaluated as little plastic as compared to the group of illite and montmorillonite clays.

### 2.1.3 Kaolin

The kaolin name derives from Kau-ling, China location from which the first samples were extracted. Kaolin is mainly composed of kaolinite. Kaolinite is a clay mineral whose chemical formula is $\text{Al}_2\text{O}_3\cdot2\text{SiO}_2\cdot2\text{H}_2\text{O}$ and has highly refractory behavior after firing (Biffi 2002).

In many situations, kaolin used in the production of porcelain tile in Brazil is obtained from the processing of kaolinite clays in order to increase the percentage of kaolinite and reduce the content of impurities. Importantly, the main characteristic of kaolin for use in ceramic masses for the manufacture of porcelain tiles is its whiteness. This feature directly influences the market value of the product as it is a great indicator of the material purity, and the higher its whiteness, the brighter the appearance of the ceramic mass after firing. According to Biffi (2002) in addition to providing whiteness to the ceramic mass, kaolinite is a basic supplier of aluminum oxide ($\text{Al}_2\text{O}_3$), which during the glazing phase of the ceramic mass, becomes a regulator of the equilibrium of reactions. In fact, alumina can take part in the formation of a silicon aluminous glassy phase in association with alkaline fluxing elements or also found predominantly at the end of the firing process as mullite ($3\text{Al}_2\text{O}_3\cdot2\text{SiO}_2$), which due to its needles morphology, acts as a “skeleton” to products obtained contributing to increase the mechanical strength.

The light color of the porcelain tile samples is desired by manufacturers and is caused by the interaction of $\text{Fe}_2\text{O}_3$ and $\text{TiO}_2$ with pigments and dyes, resulting in deviations in color of glazed samples. According to Moraes et al. (2007), ceramic kaolin must have kaolinite content between 75 and 85 % and absence of minerals affecting the firing color such as hematite ($\text{Fe}_2\text{O}_3$), whose content should be less than 0.9 %, so that the whiteness index after firing is within the range from 85 to 92.
2.1.4 Feldspar

According to Biffi (2002), feldspars are defined as silicon aluminates of alkali and alkaline earth metals. From the chemical point of view, they are classified into the following types:

- Orthoclase $K(\text{AlSi}_3\text{O}_8)$ potassium feldspar;
- Albite $Na(\text{AlSi}_3\text{O}_8)$ sodium feldspar;
- Anorthite $Ca(\text{Al}_2\text{Si}_2\text{O}_8)$ calcium feldspar.

Feldspars play a fundamental role in ceramic masses for porcelain tile production. In fact, these minerals provide high vitrification and mechanical strength at the end of the firing process.

To achieve accurate water absorption and mechanical strength values, which characterize porcelain tiles, it is necessary to suppress open porosity and reduce closed porosity after firing. For this purpose, the use of fluxing raw materials in the ceramic mass is necessary, so that a liquid phase is formed before maximum firing temperature is reached. This liquid formed during firing must have adequate quantity and viscosity to promote the filling of the pores present in the ceramic body and also improve the reactivity between elements, so that the onset of sintering occurs at lower temperatures. The most characteristic behavior of this mineral is the remarkable fusibility and the formation, with other elements, of eutectic elements that enable achieving vitrification even at relatively lower temperatures. The basic element for the fluxing properties is the content of alkali in the mineral. The theoretical values of K$_2$O and Na$_2$O, respectively, in potassium and sodium feldspars are 16.9 and 11.8 %. The more the alkali content approaches the theoretical value, the higher the commercial value of the feldspar.

2.1.5 Pyrophyllite

Ceramic pyrophyllite or only pyrophyllite, is a very fine metasedimentary rock primarily composed of sericite, kaolinite and quartz, with alkali content of about 7 %, giving it fluxing characteristics. Due to its mineralogical and chemical nature, pyrophyllite shows no plastic and plastic properties and can compose up to 50 % of many ceramic masses of wet process, especially in industries in the state of São Paulo, favored for its relative abundance in the southern state (Motta et al. 1998).

2.1.6 Talc

Talc is not always present on ceramic masses to porcelain stoneware tiles. Talc is a magnesium silicate of formula $3\text{MgO} \cdot 4\text{SiO}_2 \cdot \text{H}_2\text{O}$ used in ceramic masses in varying amounts up to 4 % increased fusibility due to the formation of an eutectic
between talc and feldspar, giving better results in terms of resistance to staining and flexural modulus. P. Grosjean 1995 reports that the use of talc led to the following improvements:

- Resistance to stains (from values above 1.6 %);
- Improvement of the flexural modulus up to 30 %;
- It seems to favor the polishing operations when microporosity is minimal;
- Lowers the thermal expansion coefficient;
- Increased whiteness when in the presence of zirconium.

### 2.1.7 Quartz

Also known as ‘sand,’ quartz (SiO₂, with crystalline structure) is added to ceramic masses for porcelain tiles with the function of maintaining a siliceous skeleton in the mass when, due to the increasing temperature, the other components such as clay, kaolin, and feldspars soften. In addition, it is an important regulator of correct ratio between SiO₂ and Al₂O₃ to form mullite (3Al₂O₃·2SiO₂). It can also play the role of regulator of contraction and deformations during firing when it forms a structure of grains that are slightly attacked by the surrounding mass (Biffi 2002).

Quartz is also present in the mineralogical composition in other raw materials: clays, kaolin, and feldspars. Thus, some attention should be given to the allotropic change that occurs during heating around 573 °C, when quartz shifts from α to β, and also in the cooling step when submitted to the same temperature and the inverse transformation occurs. During these allotropic transformations, expansions and contractions occur due to changes in the crystalline structure of quartz, resulting in internal defects such as microcracks. The α-quartz has rhombohedral structure and β-quartz form has hexagonal structure.

### 2.1.8 Dosage and Grinding

Dosage is performed at the time of preparing the load to be ground. To obtain the desired product, dosage is performed based on formulation developed at the laboratory (Barbosa et al. 2008).

Depending on the moisture content (wet basis) of each of the raw materials, the wet weight required is calculated (according to the formulation) and the scale is programmed. The addition of each raw material is manually performed with mechanical shovels up to the wet weight indicated by the scale. Plastic and nonplastic materials are alternately added to facilitate the discharge operation (Barbosa et al. 2008).

In the grinding operation, there is reduction of the particle size of raw materials. The result of this ceramic processing step influences not only the properties and
behavior of the mass in the following manufacturing steps, but also the quality of
the final product after firing due to the close relationship with the sinterability of
the ceramic mass.

In most industrial plants, porcelain tile manufacturing uses the wet process
through discontinuous grinding (Ribeiro and Abrantes 2001; Barbosa et al. 2008).
The particle size distribution of raw materials specifies the size of balls in the mill
(large, medium, and small). The volume of balls is controlled by the addition of
large- and medium-sized balls based on measurements of the level every month.
This level is indirectly measured as the difference between the diameter of the mill
and void space height. The grinding efficiency is checked usually after 5 h from
start of batch production—through sieving of samples taken from the mill, and
depends on the measurement of the percentage of residue on the sieve, bulk den-
sity, and flow time (Barbosa et al. 2008).

In the grinding step by wet method, the ceramic mass is called “slip” and
viscosity must be such as to allow the total discharge from the mill by inject-
ing compressed air. To control the slurry viscosity, deflocculants are added at the
beginning of the charge of the mill. After milling, the slip follows to homogeniza-
tion and to storage tanks.

2.1.9 Spraying

Briefly, it could be assumed that spraying is the transformation of an aqueous
suspension of solid particles (slip) into dried particles by spraying the suspension
inside a heated chamber. The sprayed slip droplets acquire a spherical shape by
the action of strong pressure exerted and by the surface tension, and undergo
rapid water evaporation (Ribeiro and Abrantes 2001). Currently, mixed flow
atomizers are used, where the slip is sprayed first countercurrent to the hot
air, and then in parallel, with capacity of 16,000 kg of dust/hour. Air heated
by natural gas and forced circulation are used to extract water vapor and finer
powder (Barbosa et al. 2008).

At this stage of processing, the homogeneity of moisture and particle size of
the sprayed powder should be controlled because these variables can affect the
uniformity and regularity of the ceramic body packaging after pressing and may
cause changes in color and dimensions from one batch to another. According to
Barbosa et al. (2008), defects in the final product such as twisted square, loop,
black heart, peeling, dirty stamping, size variations, and cracks due to low
mechanical strength may be associated with these variables.

The moisture content of the atomized powder is controlled by adjusting the
inlet air temperature, pressure in the slip pump, or flow of the exhausting air
through the exchange of worn pads. Negre et al. (2000) state that variations in the
moisture content of the atomized powder is mainly responsible for small changes
in the density of the green body, and consequently, differences in the dimensions
of the fired product. The particle size is manually controlled using as measuring
devices, manual or electromechanical sieves. The atomized powder is continuously produced through conveyor belts to storage silos. The material remains in silos for at least 24 h in order to obtain homogeneous moisture.

2.1.10 Pressing

At this stage, the ceramic body is formed and assumes its final shape, taking into account the shrinkage after firing. So, as in all stages of the porcelain tile processing, the pressing stage has its importance because the positive or negative effects will be observed in the behavior of the material pressed in the following steps, and especially in the microstructure of the final product. The characteristics of the atomized powder, particularly particle size and moisture content can influence packaging during pressing. The highest possible packaging is desired in order to maximally reduce intergranular porosity, i.e., voids between particles. According to the literature, the higher the density of the green body, the greater the reactivity during firing. It could be inferred that the more particles are in contact with each other (high density values), the largest contact surface and then higher the possibilities of severe reactions during firing.

The conformation of the material is made by double-action differential uniaxial pressure. In the production of porcelain tiles, hydraulic presses with capacity of 2,000 tons with four cavities are used. The press has its own automatic pressure control and verifications are hourly performed (Barbosa et al. 2008). According to Biffi (2002), the compression pressure for the production of porcelain tiles must be between 350 and 450 kg/cm² because the density of particles must allow, during firing, oxidation of organic substances and removal of gases that are generated. Compaction pressures higher than 500 kg/cm², in some cases, can cause internal defects and thus increased porosity of the fired body.

2.1.11 Drying

Before the firing step, the green ceramic body must be dried, i.e., its moisture content should be reduced from approximately 5.5–6 % to less than 0.5 %. This is due to the very high heating rate to which pressed bodies are submitted. Moisture contents above this parameter would cause cracks, warps, and eruptions on the surface of the ceramic body due to rapid loss of water. According to Brownell (1976) and Barbosa et al. (2008), the drying operation is performed with vertical dryers by hot air circulating, which generates the required gradients, both for mass transfer and for heat transport.

The temperature at the dryer outlet, in general, ranges from 180 to 210 °C and then the pressed part is directed to glazing and decorating processes when it comes to the production of glazed and/or decorated porcelain tile. The heat transferred to
the green ceramic body assists in the drying step helps the impregnation and adhesion of the ceramic glaze, and especially the soluble salts used for decoration. In the production of polished porcelain tiles, the pressed material is not submitted to glazing and decorating steps, going straight from the dryer to the firing furnace.

2.1.12 Firing

As previously mentioned all stages of porcelain tile processing have their importance and influence the final characteristics of the product. However, the firing stage is undoubtedly the watershed of ceramic processing because the ceramic sintering, responsible for all changes in the microstructure of the material, occurs in this stage. It is also necessary to mention the vitrification phenomena through the formation of the liquid phase that flows to interstices filling the pores and when cooling, promotes the formation of a glass that results in a dense and durable body. The vitrification degree depends on temperature and cooking time, as well as on the composition of the body. The temperature at which the liquid phase is formed is reduced by adding fluxing agents such as feldspar. During firing, as the temperature increases, the ceramic body contracts, with consequent decrease in porosity, making it denser or sintered (Castro and Benthem 2013). At this stage, constriction occurs along the contact between neighboring particles. With the advancement of sintering, the pores tend to decrease and acquire more spherical shape. The sintering of clay minerals is conducted at temperature below the melting temperature, so that in general (without the use of fluxing agents), a liquid phase is present. Mass transport is achieved through diffusion of atoms from the particles within the material to constriction regions.

It should be accentuated that both sintering and vitrification occur by heating at high temperatures, but glazing cannot occur during sintering of a ceramic mass. Vitrification is directly related to the formation of the liquid phase and in the presence of elements that produce liquid phase at temperatures lower than the maximum firing temperature. These elements are commonly known as fluxes. Both phenomena result in densification and reduced porosity, favoring the mechanical properties.

Manufacturing ceramic bodies with maximum densification, extremely low water absorption, high resistance to flexure, abrasion, and stain are only achieved by adding fluxing elements to the ceramic mass, because the sintering of clays and kaolin is not enough to drastically reduce the porosity of ceramic bodies. The formation of liquid phase during heating with adequate characteristics of quantity and viscosity, minimal addition to filling the pores, anticipates and enhances chemical reactions and changes of phase and mass transport, i.e., the sinterability, and also promotes vitrification of the ceramic body. This is the difference between porcelain tiles and other ceramics classes for coating.

Another approach that should be highlighted refers to the formation of mullite from kaolinite. When heated at about 450 °C, kaolinite begins to lose structural
water, turning into metakaolin. This process occurs up to temperature of 600 °C, approximately. According to literature, with continued heating, close to 980 °C, metakaolin turns into a Si–Al spinel (2Al₂O₃·3SiO₂) and nonreactive silica. If heating reaches temperatures between 1,000 and 1,100 °C, the spinel begins to transform into orthorhombic mullite 3:2 (3Al₂O₃·2SiO₂), known as primary mullite. When the temperature exceeds 1,250 °C, the spinel disappears and the appearance of a 3:2 mullite begins to be observed at acicular or needle shape, called secondary mullite (Restrepo and Dinger 2003; Santos et al. 2006). Restrepo and Dinger (2003) reports that mullite developed from Al–Si spinel, called primary mullite, forms large blocks and lamellar crystals and little contributes to increase the mechanical strength of porcelain parts. The crystallized mullite from the amorphous aluminum silicate phase, called secondary mullite grows in the shape of needles and help strengthen the system in the same way as fibers reinforce composites. The author also reports the secondary mullite crystals in the shape of needles only form from the melt material or in the presence of liquid phase.

It should be observed that the presence of flux materials in the ceramic mass also allows the crystallization of secondary mullite at lower temperatures (from 1,100 °C), since the liquid phase necessary for mullite crystallization in acicular format will be present before the maximum firing temperature is reached and, therefore, before the merger of clay minerals (Noni et al. 2010).

Currently, for the production of porcelain tiles, horizontal roller ovens with length of about 120 m are used, as well as natural gas as fuel to generate heat are used. The oven is divided into regions along its length, each of which has a thermocouple and a servomotor as actuator to automatically modify the air/fuel ratio in the corresponding burner (Barbosa et al. 2008). According to Biffi (2002), roll ovens allow a number of adjustments performed with the aid of microprocessors, able to avoid temperature differences between the center and sides of the oven, obtaining products with great geometric features. The firing cycle of porcelain tiles decreased considerably if compared to the cycles used in the mid 1990s, time at which there was a great production expansion and marketing of porcelain tiles.

The current firing cycles used by manufacturers of porcelain tiles comprise time intervals ranging from 40 to 60 min, with holding time from 6 to 8 min at

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Fig. 2.2  Typical firing curve for porcelain tile
maximum firing temperature, between 1,190 and 1,230 °C, and according to Biffi (2002), this temperature may reach 1,250 °C for “super white” products containing refractory elements such as zirconium silicate and alumina. Figure 2.2 shows a typical firing curve of porcelain tile production, where heating, plateau, and cooling are observed.

### 2.1.13 Polishing

This step is performed only on unglazed porcelain tiles. Polishing step is an innovation in the production of ceramic tiles. Brightness is a feature quite sought by coating consumers, because besides the esthetic effect, it also facilitates cleaning.

During grinding and polishing, a layer with about 0.5–1.0 mm of thickness is removed (Oliveira 1998). However, parts submitted to polishing are more susceptible to staining because the removal of this layer exposes pores that were previously closed. According to Oliveira (1998), the existence of irregularities on the surface of a ceramic body causes particles to adhere to its surface and makes them more difficult to be removed. Relatively large pores (>60 µm) facilitate cleaning of the product but determine loss of esthetic appearance on the product surface, since these pores can be viewed with the naked eye. Other studies point out the pore diameter as the main cause for the incidence of stains on polished porcelain tiles (Arantes et al. 2001; Amorós et al. 2007). Alves and Baldo (1998) assessed which class of pore sizes was responsible for making the polished surface of the porcelain tile more susceptible to the staining phenomenon and found that parts that had the highest percentage of pores with diameters ranging from 5 to 20 µm were more susceptible to staining. The authors state that for surfaces with greater presence of pores with diameters below this range, the penetration of staining agents is more difficult, and for surfaces with greater presence of pores with diameters above this range, the removal of staining agents is facilitated.

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