Digital decoration for ceramic tiles: The effect of glazes particle size distribution on the inkjet decoration

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ABSTRACT

After the consolidation of the digital decoration technology in the ceramic tile industry, digital glazing is the next step toward a fully digital decoration line. To make this possible, a key requirement, regarding glaze suspensions characteristics, is the reduction of the actual particle size of the solids. In this context, the objective of this work is to evaluate the effect of the particle size distribution of frits used to produce a transparent glaze on the glaze layer characteristics and the ink behavior over the same. A frit was milled under different conditions. The milling process has mainly reduced the size of the larger particles, leading to narrower particle size distributions. As the distribution curves narrowed, the particles packing efficiency in the glaze layer decreased. For these more porous layers, faster ink absorption into the surface pores and a consequent more limited ink spreading were observed, which led to the production of smaller dots. Thus, changes in granulometry of the glaze raw materials affected not only the glaze layer porous structure, but also aspects of the image quality.

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Decoración digital para baldosas cerámicas: el efecto de la distribución del tamaño de partícula de los esmaltes en la decoración por inyección de tinta

RESUMEN

Después de la consolidación de la tecnología de decoración digital en la industria de las baldosas cerámicas, la esmaltemación digital es el siguiente paso hacia una línea de decoración completamente digital. Para hacer esto posible, un requisito clave, con respecto a las características de las suspensiones de esmalte, es la reducción del tamaño de partícula actual de los sólidos. En este contexto, el objetivo de este trabajo es evaluar el efecto de la distribución
del tamaño de partícula de las fritas utilizadas para producir un esmalte transparente en las características de la capa de esmalte y el comportamiento de la tinta sobre el mismo. La frita fue moida en diferentes condiciones. El proceso de molienda redujo principalmente el tamaño de las partículas más grandes, lo que lleva a distribuciones de tamaño de partícula más estrechas. A medida que las curvas de distribución se estrecharon, la eficiencia del empaquetamiento de las partículas en la capa de esmalte disminuyó. Para estas capas más porosas, se observó una absorción más rápida de la tinta en los poros de la superficie y una consiguiente propagación de la tinta más limitada, lo que condujo a la producción de puntos más pequeños. Por lo tanto, los cambios en la granulometría de las materias primas del esmalte afectaron no solo a la estructura porosa de la capa de esmalte, sino también aspectos de la calidad de la imagen.

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**Table 1 – Formulation of the glaze suspensions.**

<table>
<thead>
<tr>
<th>Raw materials</th>
<th>wt%</th>
</tr>
</thead>
<tbody>
<tr>
<td>Frit</td>
<td>53.82</td>
</tr>
<tr>
<td>Kaolin</td>
<td>5.98</td>
</tr>
<tr>
<td>Water</td>
<td>40.00</td>
</tr>
<tr>
<td>Carboxymethylcellulose (CMC)</td>
<td>0.20</td>
</tr>
</tbody>
</table>

The glazes were dried and granulated. Cylindrical green bodies with dimensions of 6 × 2 cm were prepared by pressing the granulated powder at 400 kgf/cm² in a uniaxial automatic press. The apparent density of the compacts was determined by mercury immersion. It is well known that the arrangement of the particles in the compacts does not equal the arrangement of the particles in the digital printed layer. However, it was presumed that the changes in the frits granulometry would affect the density of the green bodies similar to what would be observed for the density of the applied layer.

For the evaluation of the ink behavior over the glaze layer, the suspensions were applied on the surface of 15 × 15 cm previously fired tiles pieces. A Binl manual applicator was used to apply 0.4 mm thick layers. The samples were dried and subsequently drops of black inks were applied over the surface by a method successfully used in a previous work [12]. Ink drops were released from a fixed volume micropipette (30 μL) positioned 13 mm above the surface. The dried samples – support/glaze layer/ink drops – were fired at 1070, 1100 and 1130 °C in fast fire cycles in a laboratory electric kiln.

The ink drops spreading behaviors were investigated through the determination of the spreading ratio (SR). The SR is calculated as the ratio between the diameter of the ink dot formed over the surface (determined with image analysis software) and the diameter of the initial drop (assumed as the same as the pipette outlet port), according to the following relation [13].

\[
SR = \frac{\text{Dot diameter}}{\text{Drop diameter}}
\]  

(1)

Fig. 1 illustrates dots of different sizes formed from fixed initial diameter drops and guaranteeing a constant volume was applied. A higher SR is related to larger dots, produced when the ink spreads in a higher extent over a given substrate. In these cases, a higher ink yield may be expected since, with
Results and discussion

Fig. 2 illustrates the particle size distribution of the frits submitted to different milling conditions. The statistical parameters $D_{10}$, $D_{50}$ and $D_{90}$ represent the particle diameter when the mass percentage is equal to 10, 50 and 90% of the total, respectively. The results have shown that $D_{10}$ is very similar for all the glazes, while $D_{50}$ and $D_{90}$ decrease considerably from C to VF frits. It indicates that the milling process has produced a size reduction mainly for the larger particles, leading to narrower particle size distributions. As a result of these changes, the particles packing in the applied glaze layer over the ceramic surface should be affected.

Apparent density results for the granulated glazes compacts are presented in Table 2. From frit C to VF the density of the compacts decreases. Both the increase in the finer fraction of particles and the consequent narrowing in the particle size distribution may have contributed to this result. According to the literature, a decrease in particle size increases the superficial forces with respect to the gravitational/hydrodynamics ones [14]. It favors floc formation and leads to less efficient particles packing in suspensions and green bodies [15]. In addition, for wider particle sizes distributions, the accommodation of smaller particles in spaces between larger ones contributes to increase packing efficiency. On the other hand, when the particles have similar sizes (narrower particle sizes distribution), lower packing densities are achieved.

In Fig. 3, a linear decrease in the glaze green bodies apparent density with the difference between $D_{90}$ and $D_{10}$ is shown. It confirms that particle packing efficiency is strongly correlated to the particle size distribution of the frits, altered by

![Image of the glaze compact density](image)

**Table 2 – Apparent density of the glaze compacts.**

<table>
<thead>
<tr>
<th>Glaze</th>
<th>Density (g/cm³)</th>
</tr>
</thead>
<tbody>
<tr>
<td>C</td>
<td>1.60 ± 0.01</td>
</tr>
<tr>
<td>M</td>
<td>1.57 ± 0.01</td>
</tr>
<tr>
<td>F</td>
<td>1.53 ± 0.01</td>
</tr>
<tr>
<td>VF</td>
<td>1.51 ± 0.01</td>
</tr>
</tbody>
</table>

![Image of the particle size distribution](image)

**Fig. 2 – Particle size distribution and statistical parameters for the frits milled in the four different conditions.**
milling conditions. Although these results refer to cylindrical compacts, with the narrowing in particle size distribution of the frits, a similar density reduction is expected for the glaze layers obtained from a suspension.

Packing of particles in the glaze layer determines its porosity, which should affect the penetration of the inks applied in digital decoration into the surface. Fig. 4 presents the spreading behavior of digital inks over the surface of the four different fired glaze layers studied. These results are correlated to the apparent density obtained for the cylindrical compacts previously evaluated, taken as an indicative of the density of the glaze layers on which the inks drops were applied.

One can note that the SR decreases linearly with respect to the packing density of the glaze compacts, reduced from frit C to VF. It was confirmed, therefore, that the changes in apparent density of the glaze due to changes in the granulometry of the frits influenced the ink spreading over the surface.

A decrease in the SR with the reduction of the apparent density of the glaze compacts can be explained by its higher porosity. When an ink drop impacts a surface, its behavior is controlled by different physical processes and driven by inertial, capillary and gravitational forces [16]. For surfaces with greater porosity, the ink penetrates more quickly. Consequently, the drop dries before it spreads to its maximum extent, which leads to the formation of a smaller dot. On the other hand, more efficiently packed layers are less permeable. Then, the ink drop spreads further over the surface, forming a larger dot.

Therefore, it can be stated that changes in the frit granulometry affect the glaze layer porous structure and,
consequently, the inks absorption and spreading in the decoration stage. This, in turn, determines the diameter of the produced dots, which interferes with the ink yield and also the resolution of the printed images.

Fig. 5 shows an image of applied drops in the glaze surface, after firing. It is observed the drops have a similar circular shape.

**Conclusions**

The different milling conditions changed considerably the particle size distribution of the frits, which affected the particle packing and the porous structure of the glaze layers. From frit C to VF the fraction of coarse particles was reduced and the granulometric distribution curve narrowed. As indicated by the apparent density results for the cylinders pressed with the granulated suspension, the glaze layers produced from frits with narrower particle size distribution are less efficiently packed. The higher porosity of these layers lead to a faster ink absorption and more limited ink spreading over the surface. Consequently, smaller dots were formed, which could contribute to higher resolution of the printed images, despite a reduction in the ink yield.

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**References**


