EFFECT OF ENAMEL PARTICLE SIZE REDUCTION DURING INTERACTION WITH DIGITAL INKS.

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1. SUMMARY

Ceramic enamel confers unique properties to tile, both technical and aesthetic. Changes in any stage of the tile manufacturing process may affect the following stages. Considering the importance of the role that enamel plays, the objective of this report is to evaluate the effect of reducing the size of frit particles in transparent enamel during interaction with industrial digital inks. The results indicate that by changing particle size distribution, particle binding is modified, consequently changing the permeability of the enamel layer, affecting penetration of the ink.

2. INTRODUCTION

The ceramic tile industry has undergone several changes in recent years, among these is digital technology. Given the importance of enamelling and decoration in the manufacture of ceramic tiles, it is necessary to monitor the changes occurring with this technology, and also that enamels may be applied using this method. Accordingly, enamel particle size becomes important, since this technology requires smaller sizes than those used for traditional enamels; in order to prevent obstruction of nozzles [1]. This reduction in particle size will likely affect other characteristics of the enamelling process as well as the enamel layer. Generally, in the decorating process ink is applied on the enamel layer, and when treated it may influence absorption of the ink and, therefore, the quality of the decoration; as previously indicated in other reports, changes in the substrate on which it is applied affects its penetration and spacing [2,3].

3. MATERIALS AND METHODS

Industrial transparent frit was used, which was ground in different conditions to obtain four granulometries. The grinds were carried out in a laboratory mill as follows:

a) Dry grinding with alumina balls to obtain 10% sieve residue with mesh size of 45 µm. This sample is denominated G.

b) Dry grinding with alumina balls to obtain 3% sieve residue with mesh size of 45 µm. This sample is denominated M.

c) Dry grinding with Zirconia microspheres for 10 minutes using the sample obtained in B. This sample is denominated F.

d) Dry grinding with Zirconia microspheres for 30 minutes using the sample obtained in B. This sample is denominated MF.

Next, the samples were screened using mesh of size 125 µm. Samples are characterized by particle size analysis by means of laser diffraction technique using MATERSIZER 2000 E, MALVERN UK equipment.

Using ground frit, enamel was prepared, 90% frit, 10% kaolin, a fixed percentage of CMC, 40% water by weight with the percentage of deflocculant obtained by means of deflocculation curves. After the preparation of enamel suspensions, they were characterized using flow curves to determine their rheological behaviour. This test was performed on a Brookfield rotational viscometer, which consisted of measuring the shear stress (Pa) as a function of the shear rate gradient (1/s).

The suspensions were applied on fired ceramic substrate, 15 x 15 cm, by means of vinyl with a controlled thickness of 0.4 mm. The enamelled medium was dried in a furnace at 110ºC for a minimum of 1 hour.

In order to determine the extension index, a procedure was adopted that was used in previous research [4,5]. Two digital technology decoration inks were used on ceramic tiles, one brown and the other black. Application of the inks was carried out using a micropipette with a fixed volume of 30 µL, with a radius of aperture of 0.1297 mm. This micropipette was placed at a height of 13 mm from the surface of the medium. The pieces were taken to the furnace, left to dry and then fired at three different temperatures (1070, 1100 y 1130ºC), in a rapid firing cycle.

The extension index (EI) was measured, which is related to the permeability of the enamel layer. The EI was calculated by means of the following equation:

$$IE = \frac{D_{gf}}{D_i}$$

Where, D_{qf} is the diameter of the drop that forms on the surface and D_i is the diameter of the initial drop that comes out of the micropipette. The diameter of the spot on the fired piece is measured by analysing the images photographed using Image Pro Plus software 4.5.

4. RESULTS

Figure 1 shows the granulometric distribution curves of ground frit under different conditions together with percentile data of granulometric fractions. It may be observed that there is a significant variation in the values of D50 and D90, while D10 remained practically constant, which means that the fraction of smaller particle sizes did not vary. The variation of the thick and medium fraction indicates a narrowing of the curves as the conditions varied. G being the most open curve, followed by M, F and MF. This affects how the particles will be organized in both the suspension as well as in the layer. Actually, the pore structure affected, given that particle binding defines how they will be bound in the enamel.

The rheological behaviour of enamel suspensions shows that as the average particle size is reduced, it tends to be less Newtonian, with a tendency to be more pseudoplastic. Additionally, there is an increase in the deflocculant consumption necessary to achieve maximum particle dispersion; the point with the least suspension viscosity.

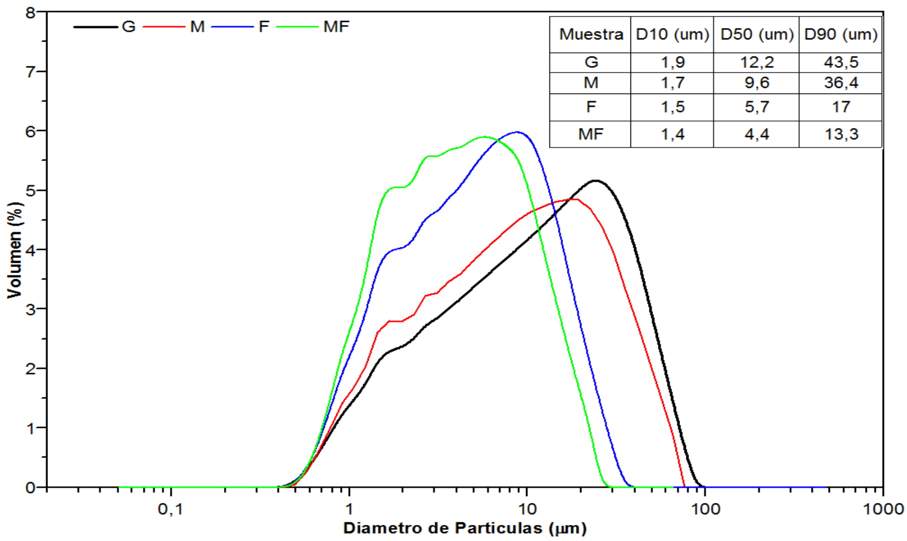

Figure 1. *Particle size distributions of the ground frit under different conditions.*

Figure 2 shows the behaviour of the inks with respect to the extension index of the drops. For both inks it is observed that the largest EI is for enamel G, which has the widest granulometric curve, and the smallest EI corresponds to enamel layer MF, with a more closed granulometric curve. This means that for a wider granulometric distribution, particle binding is more efficient; i.e., porosity is smaller, since the spaces left by coarse particles are filled by medium and small particles, therefore permeability of the enamel layer is lower, and ink does not penetrate leaving a larger drop on the surface. On the contrary, in MF, due to a more closed curve, the particles are more similar in size, binding is less efficient and, therefore, more and smaller pores, more permeable with more ink penetration, leaving a smaller drop on the surface.

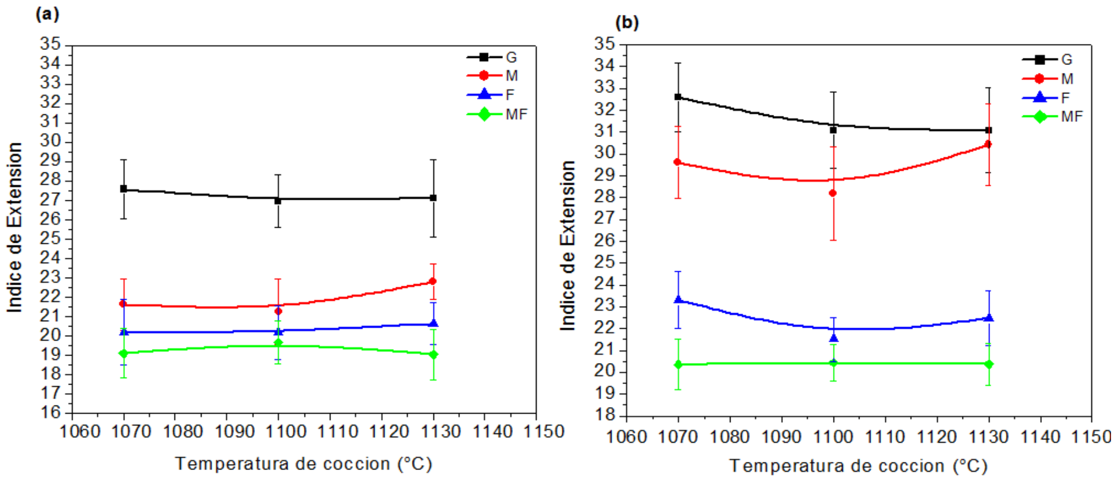

Figure 2. *Extension indexes of inks on enamel layers fired at temperatures of 1070ºC, 1100ºC and 1130ºC. (a) Brown Ink and (b) Black Ink.*

5. CONCLUSIONS

Reduction of average frit particle sizes affects enamel binding. More efficient binding occurs with wider granulometric curves.

With the application of inks it is shown that the narrowing of the size distribution curve affects pore structure and, therefore, permeability of the ink applied, given that a more permeable layer allows greater absorption, with drops of smaller sizes, which could be measured using the extension index. As the size of the drop is changing, the definition of the decoration changes; i.e., in this case there is an improvement in definition, given that smaller drops generate higher resolution per area unit.

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