# STUDY OF TONALITY VARIATION IN CERAMIC TILES CAUSED BY ROTARY DECORATING SYSTEMS

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#### ABSTRACT

The present work deals with the tonality variation of ceramic tiles decorated by the rotating technique. The study was performed on the production line of single-fired ceramic tiles, using an industrial Sincrocolor<sup>™</sup> device. The variation in ceramic ink characteristics, such as viscosity and density, and the variation of tile tonality over time were investigated. The tonality variation of the tiles was analysed from the beginning of the application process until 60min, with sampling every 20 minutes, by spectrophotometry. Samples of ceramic ink in each application time were analysed for density (pycnometry) and viscosity (flow time), and then dried and subjected to microstructural analysis (SEM and EDS). At the same time, samples of the inks were applied (0.5 mm) over engobed tiles and subjected to a 30-minute firing cycle at a maximum firing temperature of 1200°C. As a result, at 20 minutes from the beginning of the decorating process the tonality variation of the ceramic tiles is related to the variation of the ceramic ink characteristics. Initially the frit particles used in the glaze are larger (>45 $\mu$ m) than the pigment particles (<1µm). Therefore, the larger frit particles sediment over time, thus allowing the initial consumption of most of the pigment particles and thinner frit particles, resulting in diminished colour intensity with the ink application time. This effect could be avoided by an adequate milling of the frit fraction of the ink.

#### **1. INTRODUCTION**

When considering the particulate materials used in the preparation of ceramic inks, the main aspect to be evaluated is their particle size. One of the main factors contributing to the variation in tonality is the segregation of the particulate material. Usually, ceramic frits and pigments display particle sizes below  $44\mu m$  (325 mesh ASTM). Also, the particle size distribution is equally important. Considering a pigment with particles below  $5\mu m$  and a frit with particles below  $45\mu m$ , both exhibit the same fineness (D<sub>90</sub> below 325 mesh), although there may be segregation of the particulate material during the decoration process, since the finest fraction is the first to be loaded onto the roller cartridge, or segregation may occur in the ink suspension. To achieve constancy of tonality, ceramic frits and pigments should have the same fineness, as well as a similar particle size distribution.

The perfect mixing between the particulate material and the vehicle is another aspect to be considered in the dispersion of the ceramic ink. Poor homogenization of the mixture can deteriorate the print quality by increasing the tonality variation. The ceramic vehicle itself and its rheology, and the presence of moisture in the components of the ink are factors that can alter the dispersion and mixing of these components. Therefore, this work aimed to study the tonality variation in a rotary application system as a function of the application time of the ink in the decoration line. Changes in the rheological characteristics of the ceramic ink, such as flow time, density and dispersion were analysed and the best conditions for ink application and replenishment of the service tank were determined in order to avoid tonality variations.

#### **2. METHODOLOGY**

The study was conducted in a rotary decoration system using a caramel ink because the increase in ink density over time was causing tonality variation in the ceramic tiles after firing. The caramel colour was chosen because of its higher incidence of tonality variation. The ink was formulated with yellow and orange micronized pigments and water soluble vehicle, with a density of 1.45 g/cm<sup>3</sup> and 87 s flow time.

The inks were kept with stirring in the rotary decorator supply tank and the samples were collected from the return system. The tonality variation of the tiles was analysed by spectrophotometry from the beginning of the application process until 60min, with sampling every 20 minutes. Samples of the ceramic ink in each application time were analysed for density (pycnometry) and viscosity (flow time), and then dried and subjected to microstructural analysis (SEM and EDS). At the same time, samples of the inks were applied (0.5 mm layer) over engobed tiles and subjected to a 30 minutes firing cycle at maximum firing temperature of 1200°C.



#### **3. RESULTS**

The ink density remains almost constant, ranging from 1.44 g/cm<sup>3</sup> at the beginning of the decoration process to 1.45 g/cm<sup>3</sup> after the first 20 minutes, remaining stable until 40min of application and increasing again in 60min application from 1.45 g/cm<sup>3</sup> to 1.46 g/cm<sup>3</sup>. In turn, the flow time was reduced from 86s at the beginning of the process (0min) to 80s and remained stable until the end of sampling (60min). The flow time variation for ceramic inks must be between 5 and 8s.

Figure 1 shows the results of the microstructural analysis of the ink according to the application time. There is a great difference in the size of the particles between frit and pigment in the ink. There are very small particles with an average size of  $2\mu$ m and very large particles with a size greater than  $30\mu$ m or more. By microprobe (EDS) analysis, the smaller particles were identified as the pigments used in the ink and the larger particles are the frit forming the glaze.

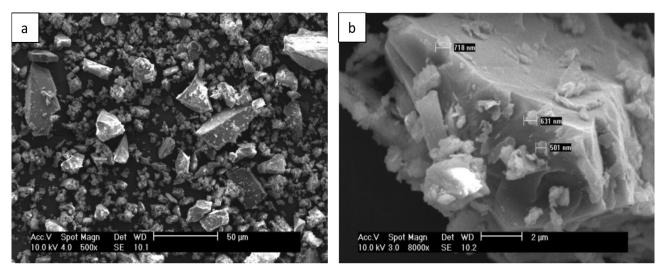


FigurE 1. Micrographs of the ink: (a) after 0 min sampling; (b) after 20 min sampling.

There is a variation in the number of particles for each time of sampling. The rheological behaviour of the ink is linked to the segregation of its solid part. According to the sampling time, segregation occurs due to the large difference in particle size between the frit particles and the pigment particles. Initially, the smaller pigment particles are consumed and with the increase of the application time the ink becomes richer in frit, thus varying its colour.

## 4. CONCLUSIONS

Regarding the study of tonality variation in rotary decoration system, the main results were:

- 1. The density of the ink remained constant during the period of study from the start of the application;
- 2. The flow time of the ink remained practically constant within the experimental error until the end of the analysis (60 min);
- 3. The microstructural analysis by SEM shows that there is a progressive decrease in the amount of ceramic pigment during the application process. Initially the pigment is well dispersed in the ink, but over the course of sampling, there is a reduction in the pigment amount in the ink. The frit presents very large particles, thereby tending to settle in the ink application tank because of the change in ink rheology over time;
- 4. Finally, there was a significant variation in the light reflection at the lower wavelengths among the ink samples, especially at the beginning of sampling (0min) with regard to the other samples (20min, 40min and 60min), showing a change in the ink characteristics from 20min sampling.

Therefore, the milling of the frit to a smaller particle size could prevent the segregation of the frit in the tank and thus the excessive consumption of pigment in the beginning of the application, resulting in less variation in tonality due to the variation of the pigment/frit ratio during the application process.