## **Ponencia ref 111**-study of ink density and screen opening as effects on tonality variation in ceramic tile decoration

Fábio Elyseu<sup>a</sup>, Nivaldo Cabral Kuhnen<sup>b</sup>, Adriano Michael Bernardin<sup>b,c</sup>

#### amb@unesc.net

# <sup>a</sup>Tecnologia em Cerâmica e Vidro, Universidade do Extremo Sul Catarinense, Criciúma, Santa Catarina, Brazil <sup>b</sup>Engenharia Química, Universidade Federal de Santa Catarina, Florianópolis, Santa Catarina, Brazil <sup>c</sup>Tecnologia em Cerâmica, Serviço Nacional de Aprendizagem Industrial, Tijucas, Santa Catarina, Brazil

The variation of silkscreen ink characteristics will cause tonality variation in tile decoration. One fast and efficient way to control tonality variation in tile decoration is the colorimetric analysis in all development steps. Several factors can influence the development of colors in tile decoration, as metamerism, illuminant, observer and object. The object is the glazed ceramic, a focus of discussion for developers of pigments, silkscreen medium and machinery, among others. Regarding pigments there are some criteria to identify if a specific variation of the pigment will affect its use, such as the color strength, given by the quantity of pigment in ink, with consequences for its use in pure form or in mixtures. Thus, this work aimed to study the effects of ink density and mesh opening on tonality variation of a ceramic glaze. A full factorial experimental design was used in the study, and the control factors were the ink density and the mesh opening; the ink was prepared according a standard ink and it was applied by flexography on a standard glaze. The analysis of variance showed that the screen opening is the factor that most influences the tonality variation of the tiles.

Keywords: pigment; ink density, silkscreen printing, tonality variation, statistical experimental design.

#### 1. Introduction:

Silkscreen printing most likely had its origins in Japan and China, and is based on the idea of stenciling. The basic principal is to place ink in some areas while eliminating ink in others to replicate an image. The ink is forced through the screen with the use of a spatula. Of all printing technologies, silkscreen printing probably remains closest to its origins with limited change over the centuries. The advance of the digital age has changed the way the screens are created. There are now machines that automate a process that was once only done by hand, but the principles of silkscreen printing are unchanged. Because the process originally used silk as the medium through which ink passed, it was called "silkscreening". Today, with a variety of media used in place of silk, it is generally referred to simply as "screen printing". Screen printing can image a wide assortment of substrates and products: fabric, vinyl, plastics, glass, ceramic and metal can all be imaged through screen printing.

Silkscreen inks are suspensions with two phases: a solid phase (colored pigment) and a liquid phase (medium). The colored pigments are oxides and the medium is generally an organic mix of polymers or mineral oils. Rheological properties of suspensions are strongly influenced by the medium and by the content of solid particles. The composition of suspensions for silkscreen inks is quite similar to the composition of ceramic glazes; the most important difference is the high oxides content of the silkscreen inks [1,2]. In the case of silkscreen ink it is fundamental to know and be able to control the density and rheological properties besides the pigments used [3-6]. The variation of silkscreen

ink rheological and/or storing characteristics will cause tonality variation in tile decoration. One fast and efficient way to control tonality variation in tile decoration is the colorimetric analysis in all development steps.

The colorimetric measurement of parameters dependent on visual comparisons with standards increases the analysis accuracy and eases the formulation and control of the desired colors with greater security, resulting in better use of ceramic pigments. A number of factors can influence the development of colors in tile decoration, as metamerism, illuminant, observer and object. The object is the glazed ceramic, a focus of discussion for developers of pigments, silkscreen medium and machinery, among others [7,8].

The formulation of inks for ceramic tile decoration has its beginnings in the development stage of the ceramic design to be produced. Designers are responsible for translating the market trends into wearable tiles, but at the same time the projects must be suitable to the production process at the most practical way possible, providing the desired effect in any produced batch, not varying the design characteristics. When formulating an ink for ceramic tile decoration, the developer should take into account the type of application which is intended: silkscreen, roller decoration, injection plotter, inkjet, and others. After setting the type of application, the developer will know the characteristics that the ink should have in order to provide a better workability of the chosen decoration system [9].

As mentioned, the characteristics of the silkscreen inks are usually related to the pigment system and the silkscreen medium, and both influence the rheological behavior of the ink suspension. Forming a solid suspension in a liquid medium, the silkscreen inks require rheological, physical and chemical characteristics as stable as possible in order to maintain color stability during tile decoration. The main aspects for evaluating an ink that meets process and post-consumer requirements are: density, viscosity and tint. These parameters are the most influential in tile decoration process using solid inks [10].

Nowadays most of the Brazilian ceramic tile industry uses the visual comparison method in order to control the tile tonality variation, which is a non adequate method. Therefore, because there are many variables related to the formulation of silkscreen inks for the ceramic industry, the focus of this work was the effect of ink density and screen opening in tonality variation of ceramic tiles using colorimetric analysis.

#### 2. Materials and methods:

One ink that had the greater degree of color difference was selected from the color palette of a ceramic tile industry after tonality variation analysis by visual perception and by spectrophotometry. In order to study the pigment behavior in silkscreen ink formulations for the decoration of ceramic tiles two variables were selected: screen opening and ink density. Therefore, ink density and screen opening were the factors of a full factorial experiment design. The analysis was made by spectrophotometry using the  $\Delta E$  parameter, the color difference coordinate, comparing the measures between the color pattern and the color sample. The pigment used was a red Se-Cd oxide and the printing medium (vehicle) was ethylene glycol. A borosilicate glaze was used with 1.5% (mass) pigment addition.

A semi-automatic screen printing machine was used to apply the ceramic ink regarding the density gradients and the screen openings according to a 3<sup>2</sup> full factorial statistical experimental design with one central point in order to study the implications of these variations in color development, Table 1. The printing machine avoided variations from the speed of application, applicator, spatula tilt, pressure, among others. This type of equipment provides a constant pressure, keeping the application homogeneous regardless of the person to perform the process.

Run	Gradient (in a 90 ASTM mesh)	Density (g/cm <sup>3</sup> )	ΔE (Judds)
1	Minor	1.20	1.1
2	Lower	1.35	2.6
3	Minor	1.50	7.2
4	Central	1.20	1.8
5	Central	1.35	1.4
6	Central	1.50	2.0
7	Major	1.20	5.3
8	Major	1.35	3.3
9	Major	1.50	4.3
10	Central	1.35	1.4

Table 1. Full factorial 3<sup>2</sup> design with a central point

Table 1 shows the entire region of the full factorial experimental design, where the studied factors are the density of the ink (g/cm<sup>3</sup>) and the screen opening gradient regarding the 90 ASTM mesh, and the levels of each factor are the minor, central and higher mesh gradient, and ink densities of 1.20g/cm<sup>3</sup>, 1.35g/cm<sup>3</sup> and 1.50g/cm<sup>3</sup>. These densities were chosen to cover a full range of density variation for flexographic and intaglio printing systems. The mesh size gradient was selected to simulate all decoration conditions used in ceramic printing.

Previously engobed and glazed tiles were silkscreened according the 3<sup>2</sup> experimental design (Table 1). After drying (110°C, 2h), five samples for each experiment were fired in a laboratory roller kiln for 21min at 1180°C maximum temperature according a single firing cycle. The solid (pigment) and liquid (screen medium) proportions were produced The fired according the experimental matrix. samples were analyzed by spectrophotometry (d8 geometry, 400 to 700nm, D65 illuminant) in order to determine the values of  $\Delta L^*$  (indication of light and dark),  $\Delta a^*$  (green to red) and  $\Delta b^*$  (blue to yellow); the  $\Delta E_{CMC}$  represents the total change in tonality. It is considered that for shiny surfaces and bright colors a  $\Delta E$  less than or equal to 0.5 Judd is imperceptible to the human eye, for darker colors and textured surfaces this difference may be 1Judd.

The spectrophotometry readings were performed according to the experimental matrix (Table 1, three variations of densities and three gradients of application).

#### 3. Results and discussion:

Table 1 also shows the results for tonality variation of the tiles according the mesh size gradient and ink density used for all 10 experiments. The results for ink density and mesh size gradient variations show the effect of the silkscreen ink printed over the glaze. The variation in ink density and mesh gradient (screen opening) changes the concentration of pigments in the ink printed over the glaze, resulting in color differences in the fired tiles regarding the  $\Delta E$  values. The results showed in Table 1 were analyzed by analysis of variance, for determining the most significant effects of the experiment, Table 2.

Table 2. Analysis of variance (AVOVA) for the cold	r differences ( $\Delta E$ )
--	------------------------------

Factor	SS	dF	MS	F test	p test		
Mesh gradient (linear)	0.67	1	0.67	0.22	0.66		
Mesh gradient (quadratic)	10.64	1	10.64	3.54	0.12		
Ink density (linear)	6.51	1	6.51	2.16	0.20		
Ink density (quadratic)	2.75	1	2.75	0.91	0.38		
Error	15.04	5	3.01				
SS <sub>total</sub>	36.02	9					

In Table 2, the factors of study are the mesh gradient and ink density; SS is the sum of squares of main effects (factors), dF are the degrees of freedom, and MS the quadratic means. F is the test of statistical significance: the higher the value, the greater the effect of the factor being studied. Finally, p is the factor of reliability of the results, where reliability is given by 100(1-p). Analyzing the data for tonality variation measured by  $\Delta E$  the most significant factor is the gradient of the mesh size (screen opening) for the quadratic model (F=3.54), and the results have a statistical reliability of 88% (p=0.12). The results have a coefficient of only 53% of the global model analysis, Figure 1.



Figure 1. Observed values versus predicted values for the adjustment to the model

To ease the analysis of the effect of the factors on the results, i.e., the effect of mesh gradient and ink density on tonality variation of the silkscreened tiles, response surfaces or contour curves can be used, Figure 2. 101 code represents the lower mesh gradient, 102 represents the central mesh and 103 the highest. Analyzing the response surface becomes clear that the smallest change in tone occurs for an ink density bellow 1.30g/cm<sup>3</sup> and the central mesh opening, with tonality values corresponding to  $\Delta E$  near 1.0.



Figure 2. Contour curve for the effect of mesh gradient and the ink density on tonality variation

The fitting equation of the contour curve of figure 2 is given by:

$$\Delta E_{\text{CMC}} = 22267.1 - 435.4x + 2.1x^2 - 124.4y + 48.3y^2$$

Where x is the ink density and y is the screen opening (mesh gradient).

It is possible to predict the tonality variation, i.e., set the  $\Delta E$  value to zero, for example. Therefore, defining a desirability contour curve for the tonality variation in function of ink density and screen opening; the result is given in Figure 3, for the Spline fit method.



Figure 3. Desirability contour curve for the effect of mesh gradient and the ink density on tonality variation (Spline fit method)

Using the desirability contour curve the analysis changes: the tonality variation is null ( $\Delta E=0$ ) for the small screen opening (smaller mesh gradient) and for smaller ink densities, Figure 3.

#### 4. Conclusions:

The variation in ink density and mesh size gradient (screen opening) changes the concentration of pigments in the ink printed over the glaze, resulting in color differences in the fired tiles regarding the  $\Delta E$  values. The most significant factor is the gradient of the mesh size for the quadratic model with a statistical reliability of 88%. The smallest change in tone occurs for an ink density bellow 1.30g/cm<sup>3</sup> and the central mesh opening, with tonality values corresponding to  $\Delta E$  near 1.0.

However, it is possible to predict the tonality variation. Using a desirability function the tonality variation can be set to zero ( $\Delta E=0$ ) in this study for the small screen opening (smaller mesh gradient) and for smaller ink densities.

### 5. References:

[1] Palmonari, C., Tenaglia, A., Rastelli, E., Albertazzi, A., Fornaciari R. Rheological study to test a new formulation of silkscreen ink. Bol. Soc. Esp. Cerám. Vidrio, 39, 5, 627-630, 2000.

[2] La smaltatura. Società Italiana per la Ceramica Assiceram. Faenza Editrice S.p.A., 1979.

[3] Prampolini, P. Le paste da serigrafia. In: La reologia dei materiali ceramici tradizionali. Ed. Pozzi P. y Galassi, C. Faenza Editrice, p.187-203, 1994.

[4] Guarnieri, G. La serigrafía. Faenza Editrice S.p.A., 1980.

[5] Schramm, G. A practical approach to rheology and rheometry. Gebrueder Haake, 1994.

[6] Albertazzi, A., Rastelli, E. Valutazione della tissotropia e del limite di scorrimento di barbottine ceramiche industriali con viscosimetro rotazionale. Cer. Acta, 9, 4, 5-11, 1997.

[7] Galassi, C., Rastelli, E., Lapasin, R. Time dependent properties of ceramic suspensions. Cer. Trans.: Science, Technology and Applications of Colloidal Suspensions, v.54.

[8] Moreno, A. Adecuación de las propiedades de tintas y esmaltes a los sistemas de aplicación y técnicas decorativas. Castellón: Qualicer 2000, 2000.

[9] Lazaro, V., Paya, M., Garcia, M. Control del proceso de transmisión de información gráfica de la decoración serigráfica. Castellón: Qualicer 2000, 2000.

[10] Escribano, P., Carda, J.B., Cordoncillo, E. Esmaltes y pigmentos cerâmicos. Enciclopedia Cerámica, Tomo I, Faenza Editrice Ibérica, 2001.