FeO, ZnO and CuO application as pigments in ceramic compounds

Aplicación de FeO, ZnO y CuO como pigmentos en compuestos cerámicos

Karol Roa-Bohórquez[1]
Ricardo Paredes-Roa[2]
Luis Lara-González[3]
Gabriel Peña-Rodríguez[4]

[1] Universidad Pedagógica y Tecnológica de Colombia (Colombia); email: karol.roa@uptc.edu.co; ORCID: http://orcid.org/0000-0001-8696-2232

[2] Universidad Pedagógica y Tecnológica de Colombia (Colombia); email: ricardo.paredes@uptc.edu.co; ORCID: http://orcid.org/0000-0002-9172-8763

[3] Universidad Pedagógica y Tecnológica de Colombia (Colombia); email: luisangel.lara@uptc.edu.co; ORCID: http://orcid.org/0000-0002-2849-0174

[4] Universidad Francisco de Paula Santander (Colombia); email: ggabrielp@yahoo.com; ORCID: http://orcid.org/0000-0002-7114-9174

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Abstract

Among the most relevant problems of the pottery industry in Boyacá, Colombia is the absence of a range of uniform shades in their final products. The color, being an easily observable physical property, becomes a distinctive criterion for potential customers seeking a differentiating and cutting-edge product within a constantly changing environment. For that reason, the present work proposed a ceramic compound elaborated from montmorillonite clays and metal oxides such as FeO, CuO, and ZnO. The clays were characterized by X-Ray Fluorescence (XRF) and according to the chemical composition of each clay, the concentrations of metal oxides were determined as coloring agents, which varied from 1 to 8 % (by weight). The ceramic prototypes were shaped by a uniaxial pressing method at a constant compaction pressure of 15 MPa and then sintered at 950 °C for 2 h. RGB histograms were used to analyze the color of the specimens, the results showed significant changes in the pigmentation of the ceramic compound reporting red, brown and white tone. This makes viable the use of these oxides as thermosetting pigments in the production of ceramic products such as roof tiles, tablets, and tiles among other ceramic designs, which today can only be found in advanced ceramic shops.

Keywords: ceramic pigments; RGB histograms; innovation; ceramics; pottery industry.

Resumen

Entre los problemas más relevantes de la industria alfarera de Boyacá, Colombia se encuentra la ausencia de una gama de tonalidades uniformes de sus productos finales. El color, por tratarse de una propiedad física fácilmente observable, se convierte en un criterio distintivo para clientes potenciales que buscan un producto diferenciador y a la vanguardia dentro de un medio en constante cambio. Por tal razón, el presente trabajo propuso un compuesto cerámico elaborado a partir de arcillas montmorillonitas y óxidos metálicos tales como FeO, CuO y ZnO. Las arcillas se caracterizaron por Fluorescencia de Rayos X (FRX) y de acuerdo a la composición química de cada arcilla, se determinaron las concentraciones de los óxidos metálicos como agentes colorantes, los cuales variaron de 1 a 8 % (en peso). Los prototipos cerámicos se conformaron por el método de prensado uniaxial a una presión de compactación constante de 15 MPa posteriormente se sinterizaron a 950 ºC durante 2 h. Mediante histogramas RGB se realizaron los análisis de color de los especímenes, los resultados mostraron cambios significativos en la pigmentación del compuesto cerámico reportando tono rojo, café y blanco. Lo anterior, hace viable la utilización de estos óxidos como pigmentos termoestables en la elaboración de productos cerámicos tales como: tejas, tabletas y baldosas, entre otros diseños cerámicos que hoy en día solo se pueden encontrar en tiendas de cerámica avanzada.

Palabras clave: pigmentos cerámicos; histogramas RGB; innovación; cerámicos; industria alfarera.

Introduction

The manufacture of ceramics in the Cundiboyacense region in Colombia dates from 1300 BC. (Prieto, 2010), with the elaboration of clay pots and other utensils for daily use by the *Muiscas*. Then, with the arrival of the Spaniards, the pottery evolved noticeably due to the use of ovens for the firing of the clay with which bricks were made for the construction of houses.

The current technique of manufacturing common bricks is carried out with the combination of red clays (in most cases illite and montmorillonites) (González; García, 1966) and degreasers (siliceous sands), elements that fulfill the function of decrease the plasticity of the mixture and contraction (Amado; Villafrades; Tuta, 2011). Once the raw materials are homogenized, water is added for molding and the pieces are sintered at different temperatures depending on the clay and the use of the pieces (Torres; Hernández; Paredes, 2012). As a result, according to NTC 4205-1 (ICONTEC, 2009), products with different structural characteristics are obtained such as mechanical resistance between 3-14 MPa and water absorption percentages below 17 %.

Pottery has become one of the sources of livelihood in the region and also an activity that generates environmental problems due to the furnaces used (Rojas; Carreño; Gómez, 2015). These ovens were highly polluting and inefficient. However, today these factors have been mitigated with technological reconversion (Quijano; Díez-Silva; Montes-Guerra; Castro-Silva, 2014), making investments in furnaces with greater combustion efficiencies and lower emissions into the atmosphere. However, although the pottery companies made an important technological advance, the technique has remained intact for more than a century (Simbaqueba, 1958), which has prevented innovating in important aspects of product design such as color.

The color has been one of the most representative problems in the pottery industry, the reason is the complexity that occurs in the modification of the hue of the clays, due to the lack of raw materials that serve as thermostable and insoluble pigments (Monrós; Badenes; García; Tena, 2003) and keep their properties in the original matrix after the sintering process.

Thus, the tonality on the brick surface depends on the chemical composition and its sintering process (Gippini, 1966; Fernández, 1998; Guerrero; Espinel; Acevedo; Guillermo, 2017). Therefore, the methods are known by the majority of brick companies to modify the color, consist of raising the baking temperature in the oven, maintaining it for long periods (approximately 24 hours) or making a mixture of clays (Patent No. 348,443, 1886; Betancourt; Martirena; Day; Díaz, 2007). The first two methods generate multiple unwanted side effects in the final product such as chipping, fragility, black hearts, efflorescence, cost overruns and inventories in the process (Roa-Bohórquez; Paredes-Roa; Lara-González, 2018). Likewise, effects on the environment are generated due to the greater use of coal, a non-renewable resource that increases air pollution levels. In addition, the mixture of clays requires a variety of special minerals and in the Cundiboyacense region, it is difficult to find clays of this type.

Among traditional color measurement systems, RGB is one of the most used based on the additive synthesis model. This model brings together the three primary colors (red, green and blue) for color representation through RGB receivers, such as cameras, scanners, sensors, among others.

Therefore, the present study seeks to provide a solution to the problem posed by applying ceramic pigments that are generally used in kaolinitic clays and not in common clays (montmorillonites), in order to generate a range of shades on the surface of the brick and diversify the current production, since not only can bricks for the facade be manufactured, tablets, tiles, among other ceramic designs, which today can be found in advanced ceramic stores.

Materials and methods

Raw materials and sample preparation

Two clay samples were collected through a manual sampling of the cone and cracking, according to the guidelines of ASTM C 702 (2011). This procedure was carried out at a site located at kilometer 3 via Sogamoso Morcá, in the department of Boyacá, Colombia.

To achieve the range of shades, three commonly used metal oxides were selected: iron oxide Fe_2O_3 (Sigma Aldrich, 95 %), zinc oxide ZnO (Sigma Aldrich, 99 %) and copper oxide CuO (Sigma Aldrich, 95 %). They are classified as synthetic inorganic pigments for iron oxide and opaque type for zinc and copper oxides (see Figure 1).

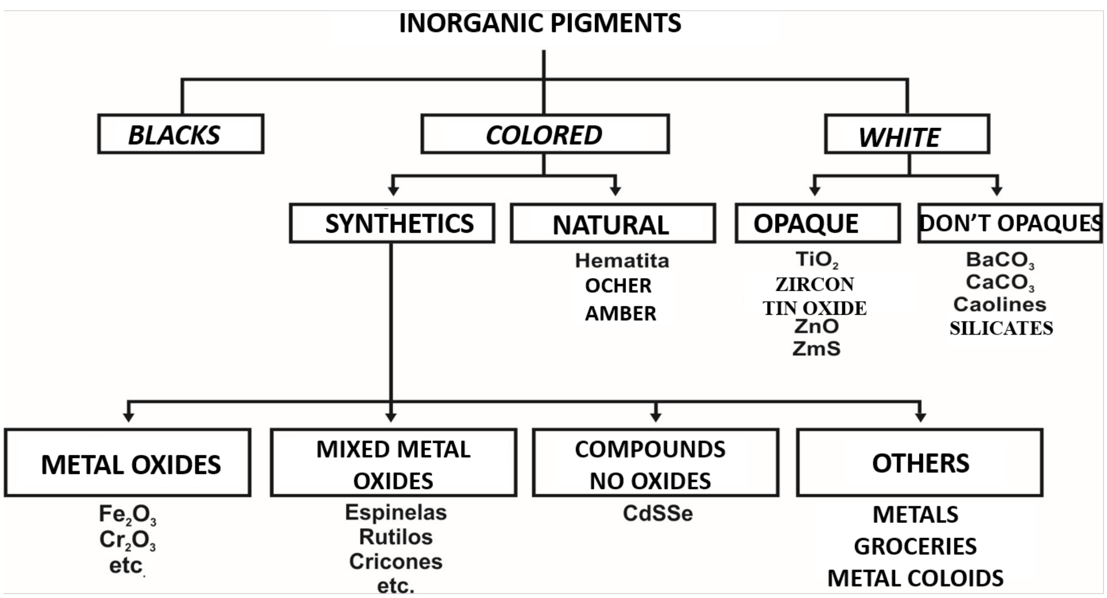

Figure 1. Classification of inorganic pigments according to the desired color
Source: Monrós *et al.*, (2003).

The mixture used for the preparation of the specimens consisted of 2, 4, 6 and 8 % iron oxide for the clay sample 2; 1, 2, 3 and 4 % zinc oxide and 4 % copper oxide, for both oxides clay samples 1 and 2 were used. Prototypes were obtained in the form of circular membranes by the uniaxial pressing method, handling a constant compaction pressure of 15 MPa. Then, the membranes were subjected to a drying process at 100 ± 5 $^{o\circ}$C for 24 hours until a constant mass was reached.

The sintering process was carried out in a Thermolyne electric muffle ref. F6018 Thermo Scientific brand, which was programmed to reach two sintering ramps at 600 and 950 °C, with a heating rate of 5 °C/min. Each temperature was kept constant for an interval of 2 h (see Figure 2).

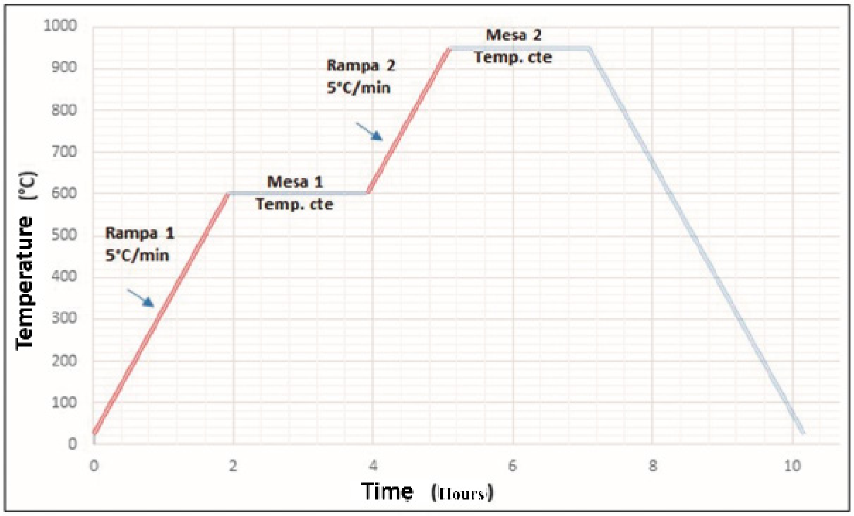

Figure 2. Sintering curve used to obtain ceramic membranes
Source: self-made.

The color analysis in each sintered membrane was performed with the public domain digital image processing software ImageJ. Prior to the study, constant lighting photography boxes with 1000 W bulbs were manufactured and the following standard parameters were taken: ISO 100 for no noise generation, 1.5 aperture and a shutter speed of 1/2000 s. The camera used in the studio is a Nikon d5300 reference.

X-ray Fluorescence (FRX)

X-ray Fluorescence (FRX) determined the chemical composition of the clays in a PANalytical MiniPal 2 brand equipment. Prior to analysis, each sample was crushed to a granulometry mesh # 200 ASTM (0.08 mm). The composition spectra obtained are presented in Figure 3. According to the semi-quantitative analysis, both clay samples revealed that the most abundant compound is silicon oxide (SiO_2), with a content of 61.8 % for sample 1 and 56, 1 % for sample 2 (Table 1). After silicon oxide, alumina (Al_2O_3) was characterized by constituting an important percentage in the samples with 29 and 22 %, respectively.

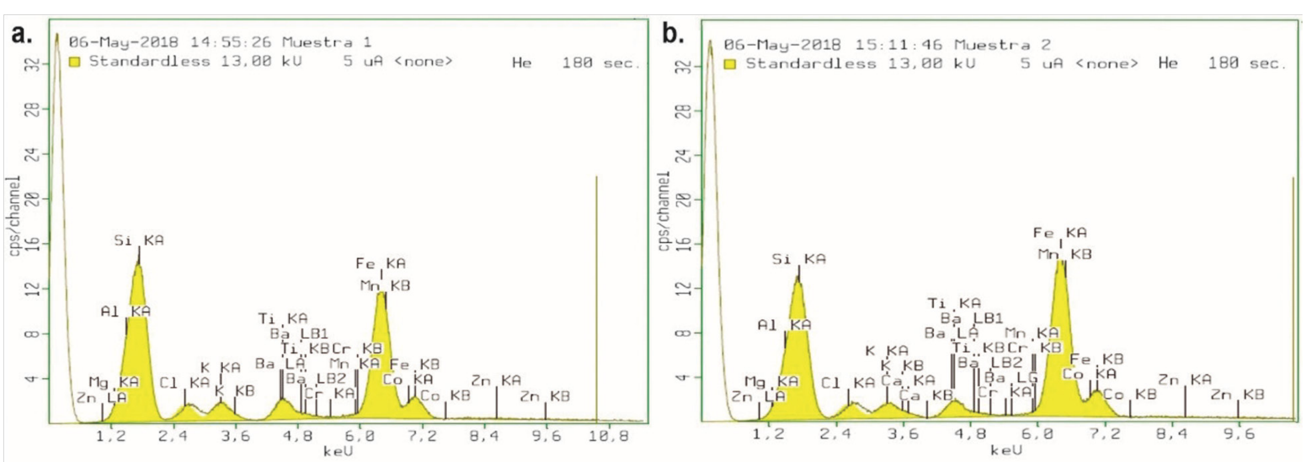

Figure 3. X-ray fluorescence spectra of clay powders
a). Sample 1 **b).** Sample 2
Source: self-made.

Table 1.
Chemical composition of clay samples understudy

Sample	Chemical component (%)										
	Al_2O_3	SiO_2	Fe_2O_3	ZnO	TiO_2	CuO	K_2O	MnO	Cl	MgO	BaO
Clay 1	29	61,8	4,57	0,05	0,9	0,03	1,3	0,09	1,1	0,5	0 , 3
Clay 2	22	56,1	12,7	0,08	1,3	0,01	2,5	0,19	2,8	0,9	0 , 4

Source: self-made.

As reported by Alvarado (1985), the molar ratio of silica and aluminum oxides allows clays to be classified as kaolinites, elites or montmorillonites. In this case, the SiO_2/Al_2O_3 molar ratio in both samples is 2: 1, which classifies them as montmorillonite clays. This type of clays is characterized by being chemically composed of SiO_2 with percentages that vary between 50 and 65 % and Al_2O_3 with participation between 10 and 25 % (Bailey, 2013). In this case, sample 1 contains alumina with a percentage greater than 25 %, however, it is not classified as clay with refractory properties, which comprise compositions greater than 40 % aluminum oxide (Díaz, 2015; Sánchez; Orozco; Peñaloza, 2014).

On the other hand, it is observed that the Fe_2O_3 and TiO_2 coloring oxides are present in high concentrations in the clay sample 2, with percentages of 12.7 for iron oxide and 1.3 % for titanium dioxide. According to the above, when the contribution of both compounds exceeds 1 %, the clays will not have a white hue after the sintering process, as reported by Avgustinik (1983), Prada (2015) and Zuluaga *et al.*, (2016). When comparing the percentages found in sample 1 (4.57 % Fe_2O_3 and 0.9 % TiO_2), it can be affirmed that with the clay sample 2 more reddish tones will be obtained in the final product (Rodríguez; Rivera; Aza, 1999). For this reason, sample 2 is selected for the application of Fe_2O_3, because lower concentrations of the metal oxide will be required to achieve uniform red tones on the surface and, therefore, will represent a lower cost for its application.

Clays reported the presence of other compounds such as K_2O, MnO, Cl, MgO, and BaO, however, taking into account their contribution, they are characterized by traces in the clay ore. In the case of the ZnO and CuO oxides, their participation in both samples is low, for this reason, the application of these metal oxides in the clays is done in both samples 1 and 2 and thus observe their behavior.

Results and Discussion

RGB color analysis

Application of iron oxide (Fe_2O_3)

In Figure 4, four sintered specimens (a, b, c and d) with iron oxide application in concentrations of 2, 4, 6 and 8 %, respectively, are observed. The clay used as the matrix of the compound belonged to sample 2 because it contained the highest concentration of this oxide. Thus, the influence of iron oxide on the reddish hue of the surface is observed, checking that described by Katsuki and Komarneni (2003), who also used commercial Fe2O3 powders as a ceramic pigment in white porcelain plates.

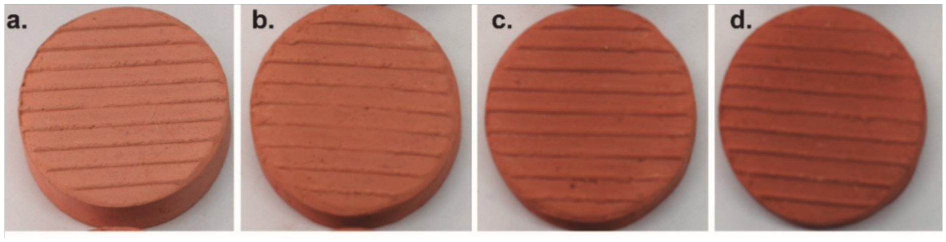

Figure 4. Sintered ceramic specimens with sample 2 of clay and iron oxide
a). 2 % Fe_2O_3 **b).** 4 % Fe_2O_3 **c).** 6 % Fe_2O_3 **d).** 8 % Fe_2O_3
Source: self-made.

To determine the coloration with greater precision, RGB color histograms were performed for the four membranes with ImageJ software (Figure 5). This histogram model is based on the additive synthesis of colors, which allows characterizing a color in the three primary colors.

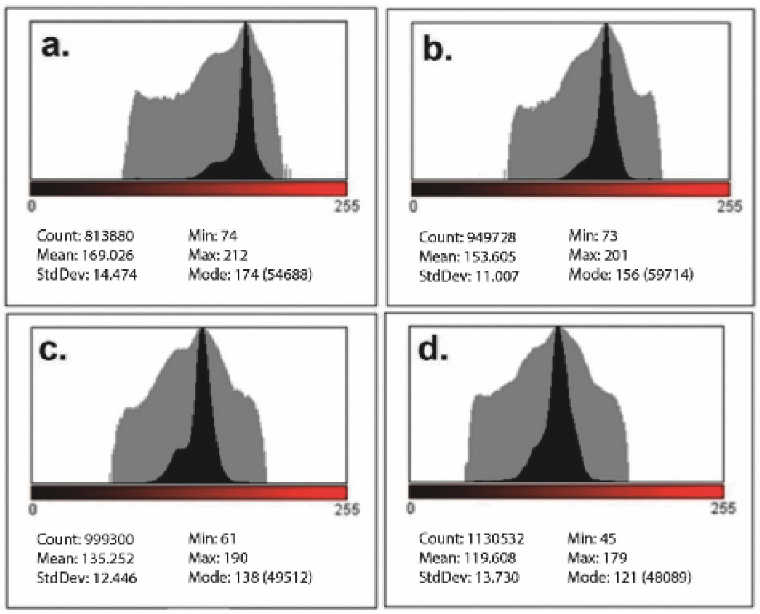

Figure 5. RGB color histograms of sintered ceramic specimens
a). 2 % de Fe_2O_3 **b).** 4 % de Fe_2O_3 **c).** 6 % de Fe_2O_3 **d).** 8 % de Fe_2O_3
Source: self-made.

For the iron oxide, the RGB analysis of the red channel was carried out, because it is the saturated channel in the histograms (Figure 5). It is observed that the *min* parameter decreases as the concentration of the oxide increases, which leads to the darkening of the color. In the four histograms, the darkest color was reported by the membrane with 8 % of Fe_2O_3 (Figure 5d), since it evidenced the lowest *mode* parameter with a value of 121.

Zinc Oxide Application (ZnO)

Because the concentrations of zinc oxide reported by the X-ray fluorescence in each of the samples were not representative (they do not exceed 1 %), the application of this oxide was carried out in both samples. Figure 6 shows the membranes a, b, c, and d, formed with the clay sample 1 in percentages of 1, 2, 3 and 4 % pigment, respectively, where it is observed that with the application of zinc oxide it also generates resistance to corrosion and UV rays, as reported by Kiomarsipour; Razavi; Ghani and Kioumarsipour (2013).

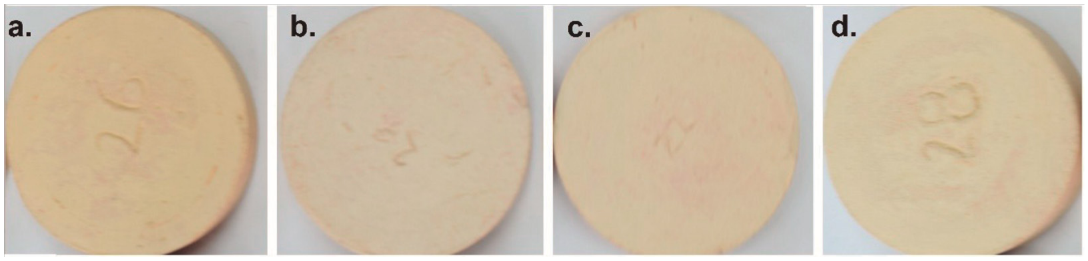

Figure 6. Sintered ceramic specimens with sample 1 of clay and zinc oxide
a). 1 % ZnO **b).** 2 % ZnO **c).** 3 % ZnO **d).** 4 % ZnO
Source: self-made.

The RGB color histograms for the four membranes of Figure 6 are presented in Figure 7.

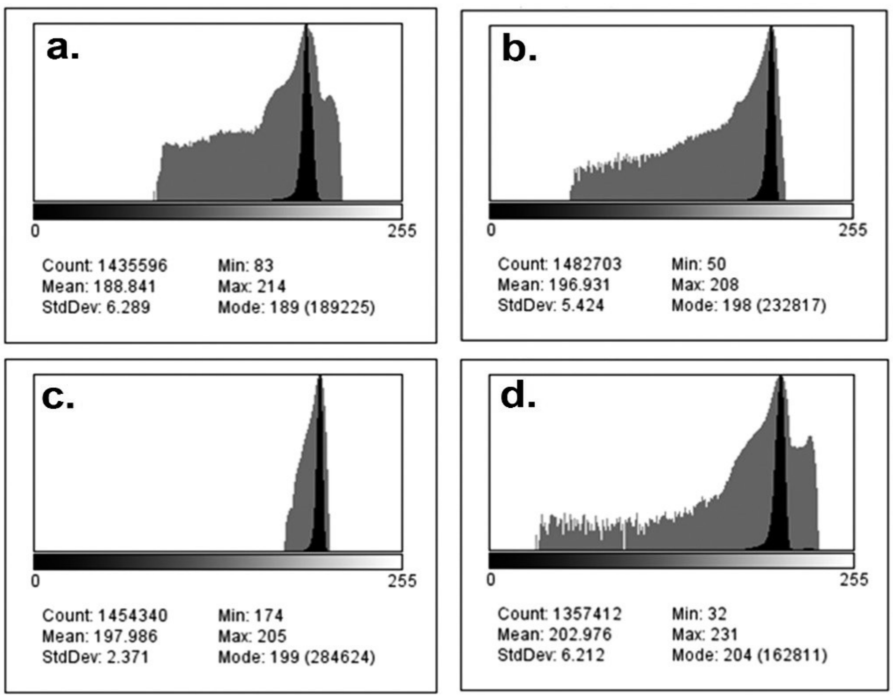

Figure 7. RGB color histograms of sintered ceramic specimens
a). 1 % de ZnO **b).** 2 % de ZnO **c).** 3 % de ZnO **d).** 4 % de ZnO
Source: self-made.

For zinc oxide, the indispensable RGB analysis belongs to the white channel, because it is the saturated channel. For the membranes of Figure 6, the histograms show that the *mode* parameter increases proportionally to the concentration of the oxide, that is, the peaks of each curve are getting closer and closer to the white color (reference *mode*: 255). Of the four histograms, the lightest color with a tendency to opaque white was reported by the sample with 4 % zinc (Figure 7d) with a model parameter of 204 (closest to 255).

In Figure 8 the membranes a, b, c, and d represent the membranes made from clay 2 with percentages of ZnO of 1, 2, 3 and 4 %, respectively. At first glance, it can be seen that sample 2 did not show the same shades as the membranes of sample 1 since the same pigment concentration was managed in both. This is due to the high content of Fe_2O_3 of sample 2, demonstrating its influence as a generating oxide of a high reddish hue with greater difficulty in modifying.

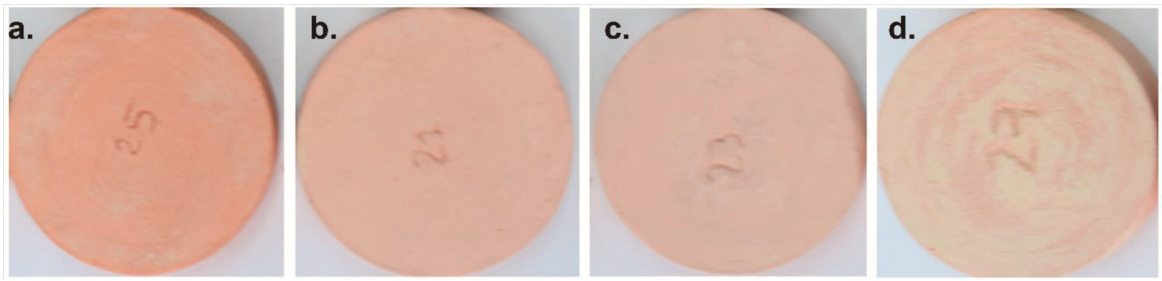

Figure 8. Sintered ceramic specimens with sample 2 of clay and zinc oxide
a). 1 % ZnO **b).** 2 % ZnO **c).** 3 % ZnO **d).** 4 % ZnO
Source: self-made.

The RGB color histograms are shown in Figure 9 for the four membranes of sample 2, of Figure 8. As in sample 1, the histograms report a *mode* parameter that increases proportionally with the concentration of the oxide, the peaks of each curve they approach the white color with a lower intensity. The lightest color found in the samples is exhibited by 4 % zinc oxide with a *mode* parameter of 200 (the closest to 255).

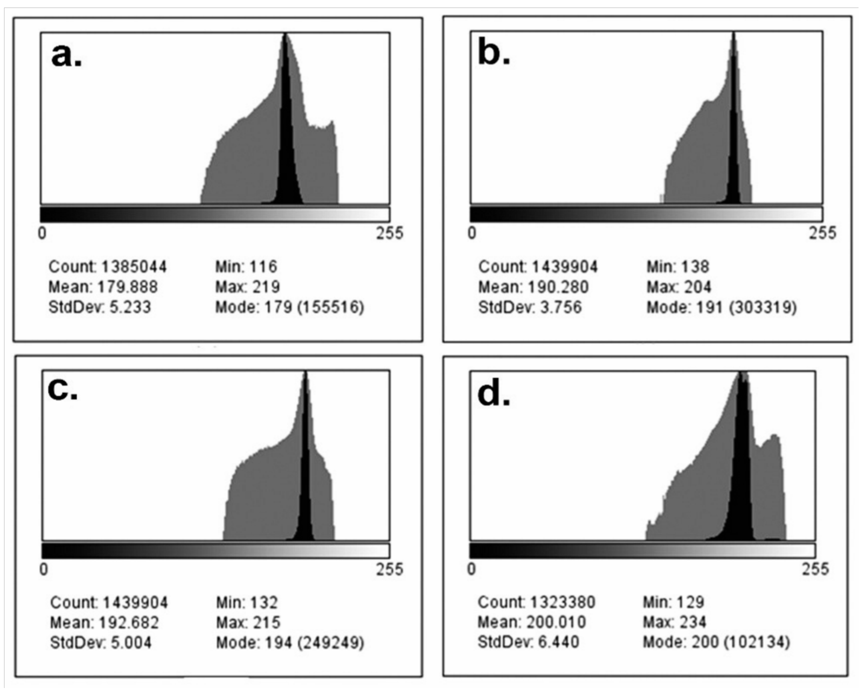

Figure 9. RGB color histograms of sintered ceramic specimens
a). 1 % de ZnO **b).** 2 % de ZnO **c).** 3 % de ZnO **d).** 4 % de ZnO
Source: self-made.

Copper oxide application (CuO)

The membranes presented in Figure 10 represent the specimens made from the samples of clays 1 and 2 with a concentration of 4 % CuO. According to the work done by Gargori; Galindo; Polish Fas and Monrós (2014), copper oxide additions between 3 % and 5 % manage to modify the tonality of clay materials, as well as to favorably increase their absorption and electrical conductivity, as well as their thermal conductivity (Cheng; Chai; Zhang, 2014). It is observed that the specimen made from the clay sample 2 resulted in a brown hue stronger with the same amount of pigment as the test tube elaborate with the clay sample 1. This change can be attributed to the high content of Fe_2O_3 in this clay, which favors this type of tones.

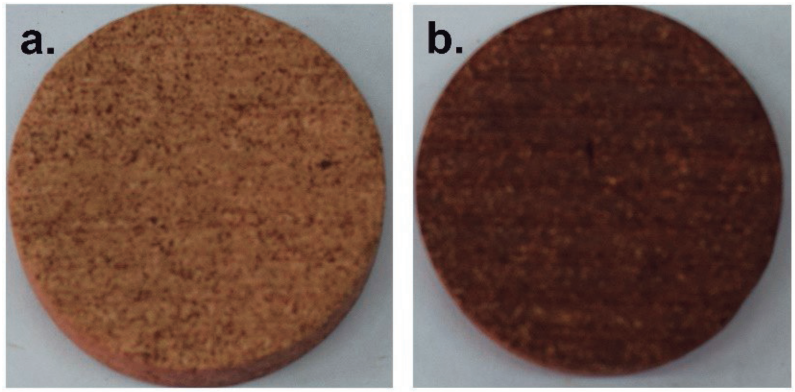

Figure 10. Sintered ceramic specimens with both clay and copper oxide samples
a). Sample 1 of clay and 4%CuO **b).** Sample 2 of clay with a high content of Fe_2O_3 and 4 % CuO
Source: self-made.

The RGB color histograms for the membranes of samples 1 and 2 are presented in Figure 11. For copper oxide (CuO) the RGB analysis was carried out with the red and blue channel because they are the primary colors to form the brown tone. When comparing the two histograms of the red channel, it is evident that the mean or *mode* parameter of the sample 2 membrane (figure 11c) tends more to the left, that is, towards the black color, reporting a *mode* of 54. On the other hand, in sample 1 it obtained 99 (Figure 11a), a value that is located further from the reference 0. In the same way, it happened with the channel or blue color of the two samples, where sample 2 (Figure 11d) is closer to the black reference color (*mode* 20) than sample 1 (Figure 11b) which obtained a value of 42.

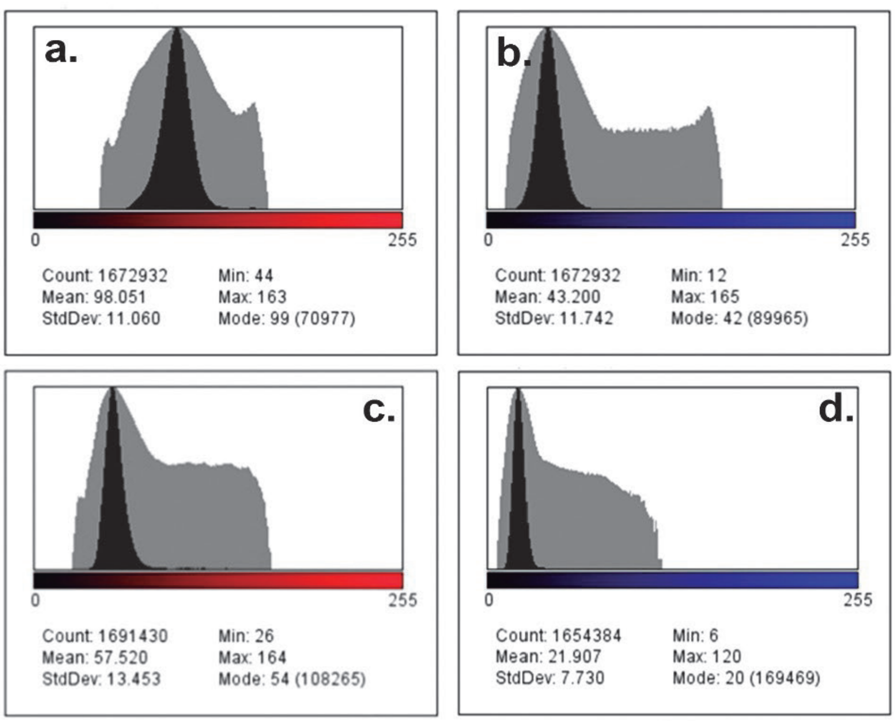

Figure 11. RGB color histograms of sintered ceramic specimens
a–b). Red and blue channels show 1 with 4 % CuO
***c–d*).** Red and blue channels show 2 with 4 % CuO
Source: self-made.

Conclusions

It is possible to use synthetic oxides to color the ceramic materials, helping to create new products and facilitating innovation within the pottery sector, as well as in the standardization of their raw materials and final products.

With the use of oxides, the hue of common clays can be transformed, however, the characterization of the raw materials is necessary because it is the fundamental part of the process. Its chemical composition significantly influences the choice of oxides and amounts to be added to generate ranges of different shades.

The application of oxides influences the sintering processes of ceramic materials, taking into account that it can replace the practice of increasing temperatures and prolonging cooking time and, in this way, reducing process times.

Zinc oxide generates a new range of light tones for common clays, which allows the manufacture of tablets and bricks for this color facade. These products are nowadays widely used in the construction of houses and for the most part, they can only be manufactured from special clays such as kaolin.

To achieve eye-catching colors and textures for customers, the application of copper oxide must be carried out, whose hue varies according to the clay used and the content of other oxides such as iron oxide.

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