

Al₂O₃-based pigments synthesized by the proteic sol-gel method

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Abstract: Alumina based pigments were synthesized by Proteic Sol-gel method. In this method, coconut water is employed as polymeric agent instead of the conventional alcóxide precursors. Three common chromophore metallic ions (Mn³⁺, Co³⁺ and Cr³⁺) were chosen in order to verify the method efficiency. Differential thermal analysis, thermogravimetry and XRD techniques were used to characterize the synthesis process. The colorimetric characterization was done according to the CIE-L*a*b* 1976 norm which is recommended by the CIE (International Commission on Illumination). The synthesized pigments presented intense and uniform colors in accordance to the literature results for each chromophore metallic ion.

The proteic sol gel technique is a promising method to obtain nanostructured powders in several ways⁽¹⁾. In this method, coconut water is employed as polymeric agent instead of the conventional alcóxide precursors. Therefore, in this work, we have studied the synthesis and characterization of the Al₂O₃-based pigments synthesized by the proteic sol-gel method. Three common chromophore metallic ions (Mn, Co and Cr) were chosen in order to verify the efficiency of the synthesis method. Thermal analysis, structural and colorimetric characterizations were also done.

Figure 1 presents the XRD patterns as a function of the calcination temperature of the AlMn samples. As can be seen, an amorphous phase was observed up to 700 °C. At 800 °C the γ -Al₂O₃ phase is crystallized. The phases γ -Al₂O₃ to θ -Al₂O₃ and α -Al₂O₃ are observed above this temperature. The powders were calcined at different temperatures and the produced pigments were characterized according to the CIE-L*a*b* 1976 norm, as described above. Table I presents the chromatic coordinates (L*, a* and b*) of the synthesized pigments. To human eyes, manganese doped Al₂O₃ pigments presented pink color; cobalt doped Al₂O₃ pigments presented a shift of dark blue to blue color; and chromium doped Al₂O₃ pigments presented a shift of dark green to green color. These colors already were expected according to the literature^(2,3). The synthesized pigments also presented intense and uniform colors and good stability after glazing.

In conclusion, alumina based pigments were successfully synthesized for the first time by the proteic sol gel method. It was observed a dependence on the thermal events and alfa-beta Alumina phase transition with the chromophore ions employed. The synthesized pigments were characterized based on the CIE-LAB color space and presented intense and uniform colors in accordance to the literature results. Besides, according to our results and observations, we have found that it is possible to produce many other colors using different chromophore ions with different concentrations by the proteic sol gel method.

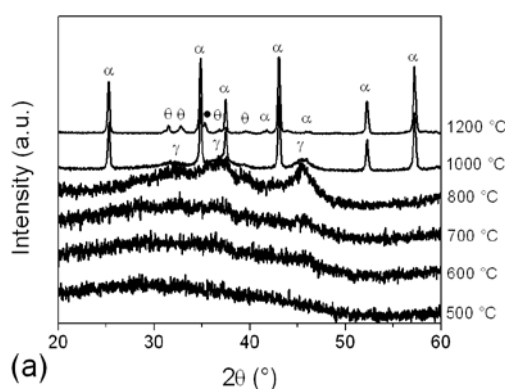


Figure 1 - XRD pattern as a function of the calcination temperature: α : α -Al₂O₃; θ : θ -Al₂O₃; γ : γ -Al₂O₃; •: KCl.

Table I - Chromatic coordinates (C.C.) of Al₂O₃-based pigments obtained by the Proteic sol gel method

| Composition | C.C. | Calcination temperature (°C) | | | | | |
|--|------|------------------------------|------|------|-------|-------|-------|
| | | 500 | 600 | 700 | 800 | 1000 | 1200 |
| Al ₂ O ₃ :1%Mn | L* | 67.6 | 66.2 | 74.5 | 74.1 | 78.6 | 89.6 |
| | a* | 6.2 | 6.8 | 8.1 | 7.5 | 12.5 | 13.0 |
| | b* | 12.5 | 13.1 | 14.9 | 15.4 | 21.1 | 17.1 |
| Al ₂ O ₃ :5%Co | L* | 57.1 | 54.3 | 54.1 | 48.9 | 51.0 | 63.7 |
| | a* | -2.8 | -3.0 | -3.0 | -4.7 | -5.9 | -4.8 |
| | b* | 2.5 | 5.3 | -5.9 | -13.0 | -55.9 | -50.1 |
| Cr _{1.23} Al _{0.77} O ₃ | L* | 19.7 | 40.0 | 42.3 | 43.4 | 49.3 | 50.7 |
| | a* | 7.7 | -7.6 | -8.5 | -5.3 | -9.6 | -7.9 |
| | b* | 23.2 | 20.0 | 20.2 | 22.7 | 16.2 | 10.2 |

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