SYNTHESIS OF ALUMINA-BASED CERAMIC PIGMENTS BY THE PECHINI METHOD

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Aluminium oxide is the most widely used oxide worldwide in ceramic applications. This widespread use is due to its low cost and the set of properties that it exhibits: high melting point, hardness and great resistance to corrosion. At the moment, one of the main ceramic pigment requirements is small particle size, to enable making designs with greater aesthetic definition. Thus, the objective of this work is to obtain alumina-based ceramic pigments with nanometric particle sizes. To produce the colours, the alumina matrix was synthesised chemically and different transition metals (Co, Mn, Fe, Cu, Ni, Cr and V) were added, yielding different colour pigments. The adopted chemical synthesis method was the Pechini method. There are two basic reactions involved in this method for precursor synthesis: (1) chelation between the metallic cationes and citric acid and (2) polyesterification with ethylene glycol in a slightly acidified solution. The metallic ions are chelated by the carboxyl groups and remain homogeneously distributed in the polymeric network. The solution is deposited on a substrate and then thermally treated to form the desired oxide. The Pechini process affords several advantages over other ceramic powder processing techniques, including low cost, good compositional homogeneity, high purity and low processing temperature. The influence of these metals on the formation and stabilisation of the different mineralogical phases and colours was determined by measuring surface area and by X-ray diffraction (XRD) of the powders, as shown in Figure 1.

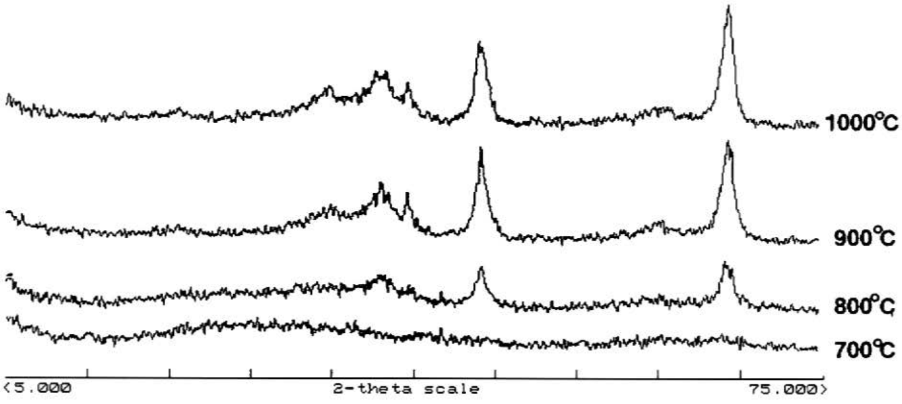

Figure 1 – Diffractograms of Al_2O_3, powders calcined at different temperatures.

Scanning electron microscopy (SEM) was used to determine the grain sizes and state of the powder agglomerate. These different ceramic pigments were applied in ceramic frits, evaluating their stability. The colour of each pigment was determined by means of the chromatic co-ordinates. The resulting pigments presented blue, pink, green and intense yellow colorations. This was achieved with low cation concentrations, unprecedented among commercially available pigments. Moreover, owing to the high resulting surface area, these pigments possess extremely small particle sizes, of a nanometric scale, which was confirmed by SEM.

Table 1 gives the molar chemical compositions of the synthesised pigments and chromatic co-ordinates L*a*b* after glazing in transparent frits.

Pigment	***Composition***	***L****	***a****	***b****
Fe8	Al_2O_3.8%	65.01	17.38	43.25
MnFe4	Al_2O_3.4%Mn.4%Fe	67.53	14.77	33.76
Mn8	Al_2O_3.8%Mn	58.73	15.27	19.66
CrFe4	Al_2O_3.4%Cr.4%Fe	67.15	11.55	32.49
Cr4	Al_2O_3.4%Cr	45.89	2.70	21.90
Cr8	Al_2O_3.8%Cr	42.58	-4.04	20.37
Ni4	Al_2O_3.4%Ni	58.53	-0.65	13.28
Ni8	Al_2O_3.8%Ni	58.37	6.51	23.77
Cu8	Al_2O_3.8%Cu	73.93	-4.33	8.17
Co2	Al_2O_3.2%Co	61.92	-1.44	-7.94
CoZr2	2%Co.2%Zr	54.84	-4.61	-19.55
Co4	Al_2O_3.4%Co	37.91	-7.41	-39.10
Co8	Al_2O_3.8%Co	36.68	-4.06	-34.24

*Table 1 – Chromatic co-ordinates L*a*b* of the synthesised pigments.*

The data set out in Table 1 were used to determine the CIELab chromatic co-ordinates in the plane a* X b*, as shown in Figure 2.

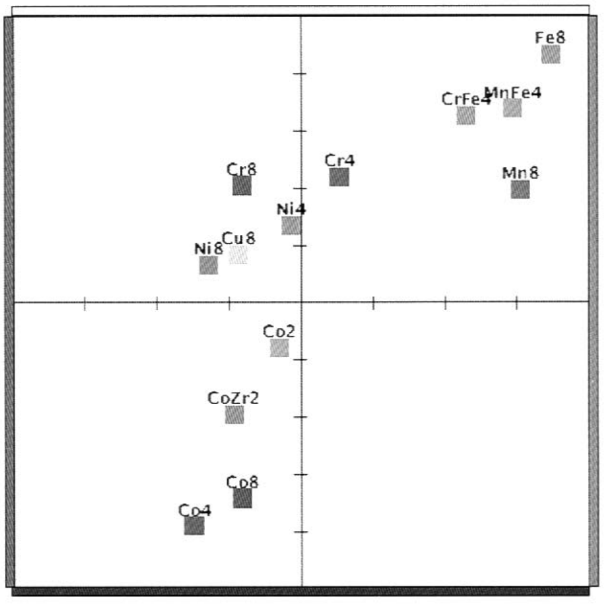

Figure 2- Chromatic co-ordinates in the plane a b* of the synthesised pigments, detailed in Table 1.*

The chemical synthesis method used in the preparation of the pigments was shown to be versatile from the point of view of synthesis ease, use of small quantities of chromophore oxides, production of stable colours in ceramic frits and small particle sizes.