SYNTHESIS AND ENCAPSULATION OF CERIUM-BASED RED PIGMENTS

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The tendency of manufacturers to exclude heavy metals from their formulations has altered the usable types of pigments, which have partly been substituted by modified (encapsulated) or completely new products that are stable in ceramic frits. The transition metal oxides of the lanthanide series, such as cerium and praseodymium, in the presence of iron oxide, present red colourings of different shades, depending on the composition.

The objective of this study was to obtain ceramic pigments based on cerium oxide with stable red shades in ceramic frits. To produce these pigments, cerium oxide was synthesised chemically, adding iron and praseodymium.

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 metal cationes and the citric acid and (2) polyesterification with the ethylene glycol in a lightly acidified solution. The metal ions are chelated by the carboxyl groups and they remain homogeneously distributed in the polymer network. The solution is deposited on a substrate and subsequently heat treated to produce the desired oxide. The Pechini process offers several advantages over other ceramic powder processing techniques, including low cost, good compositional homogeneity, high purity and low processing temperature.

	P1	P2	P3	P4
CeO_2	80	80	40	
Fe_2O_3	20	15	55	95
PrO_2		5	5	5

Table 1 – Compositions in mol % of the compositions synthesised at 800ºC.

The effect of these metals on the formation and stabilisation of the different mineralogical phases and colours was determined by surface area measurement and X-ray diffraction of the powders.

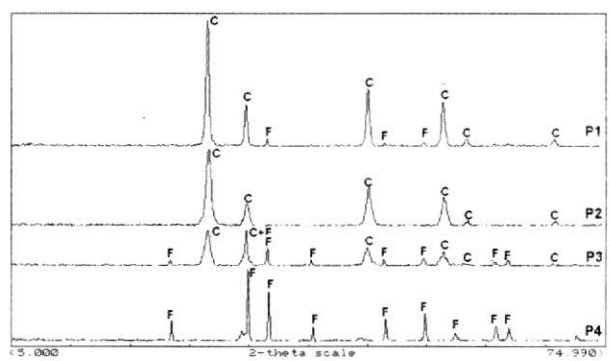

Figure 1 – X-ray diffractio of the pigments synthesised at 800ºC (P1, P2, P3 and P4 according to Table 1).

As Figure 1 shows, the praseodymium causes the cerium and iron to form a solid solution (P1→P2). Pigment P3 is a blend of iron and cerium, and P4 only contains iron.

These different ceramic pigments were applied in ceramic frits, evaluating their stability. The colour of each pigment was determined after application in crystalline frits by means of the chromatic co-ordinates, as set out in Table 2.

	P1	P1e*	P2	P3	P4
L	64.79	68.40	68.56	39.68	31.18
a	10.20	18.06	16.64	17.90	13.63
b	7.50	16.36	24.01	13.67	15.40

Table 2 – Chromatic co-ordinates of the synthesised pigments.

*Pigment P1, encapsulated with silica.

The data detailed in Table 2 were used to make the graph depicted in Figure 2. Pigment P1 kept a weak colour and was thus encapsulated.

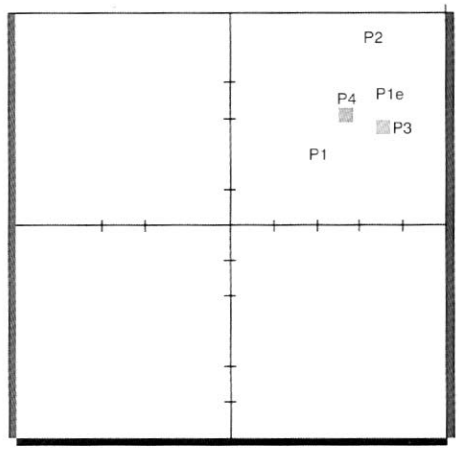

Figure 2 – Chromatic co-ordinates of the synthesised pigments.

The results demonstrate the feasibility of using these pigments, without praseodymium and encapsulated as red ceramic pigments. Furthermore, pigment P2, with praseodymium but not encapsulated, presents good colour results. Therefore, praseodymium can be considered to contribute to the stability of the pigment crystalline lattice and help maintain the colour after glazing.