SYNTHESIS AND ANALYSIS OF NANOMETRIC PIGMENTS $Li_2Co_{1-X}Ni_XTi_3O_8$ AND $Li_2Co_{1-3X/2}Fe_XTi_3O_8$

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ABSTRACT

The objective of this work has been the synthesis and characterisation of ceramic oxides with the spinel phases $Li_2Co_{1-X}Ni_xTi_3O_8$ and $Li_2Co_{1-3X/2}Fe_xTi_3O_8$ with nanometric particle size by the modified polymer precursor method (Pechini). The sought-after pigments $Li_2Co_{1-X}Ni_xTi_3O_8$ and $Li_2Co_{1-3X/2}Fe_xTi_3O_8$ were obtained above 350°C, and they display a nanometric structure. The high viscosity made it possible for monophase compounds with a high surface area to develop.

The results show the feasibility for use of the pigment in compounds that display coloration at low temperature with nanometric size.

1. INTRODUCTION

The spinel $Li_2CoTi_3O_8$[1] has recently been synthesised by the polymer precursor method and characterised by means of several techniques, thus obtaining crystalline phases at low temperatures with a nanometric-scale particle size; in the paper cited it is reported that Co^{2+} thermal stability and coloration depend on the occupied coordination. In view of this, the objective of the present study has been the synthesis and characterisation of ceramic oxides with the spinel phases $Li_2Co_{1-X}Ni_xTi_3O_8$ and $Li_2Co_{1-3X/2}Fe_xTi_3O_8$ with a nanometric particle size by the modified polymer precursor method (Pechini). The characterisation has been performed by means of thermal analysis techniques (TG), as well as XRD, BET, UV-Visible analysis, verifying the possible use of the pigments in ceramics, cosmetic, inks, and other applications.

2. EXPERIMENTAL PROCEDURE

The resin for the spinel phases $Li_2Co_{1-X}Ni_xTi_3O_8$ and $Li_2Co_{1-3X/2}Fe_xTi_3O_8$ was synthesised by the modified polymer precursor method developed by Pechini, as follows: Initially, citric acid was added in aqueous medium in the proportion of 3 moles in relation to the sum of the cations involved in the synthesis with temperatures up to 70°C; after complete dissolution this was added to the titanium citrate solution which was at the same temperature. Ethylene glycol was then immediately added in the proportion of 40/60 (% in mass) in relation to the citric acid. After solubilisation, the temperature was gradually increased up to 100°C to promote the esterification that gives rise to the polymer resin. After preparing the resin, this was subjected to a preliminary calcination with temperatures of 350°C for 2 hours; this was followed by disaggregation in an agate mortar. After the disaggregation of the material, the powder was treated at temperatures that ranged from 400°C to 700°C, at a rate of about 10°C/min in air atmosphere for 1 hour.

3. RESULTS AND DISCUSSION

Figure 1

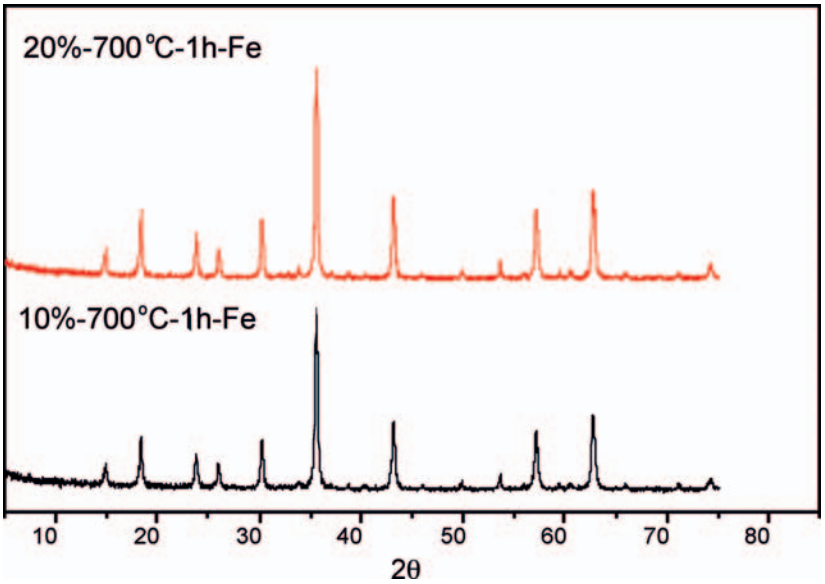

Figure 2

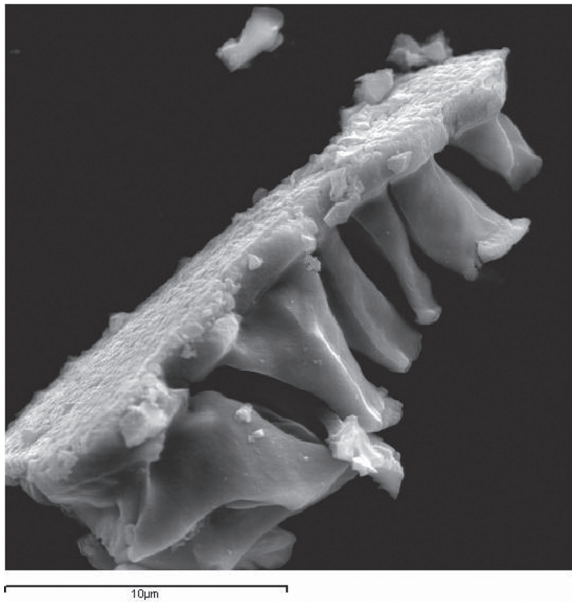

Figure 3

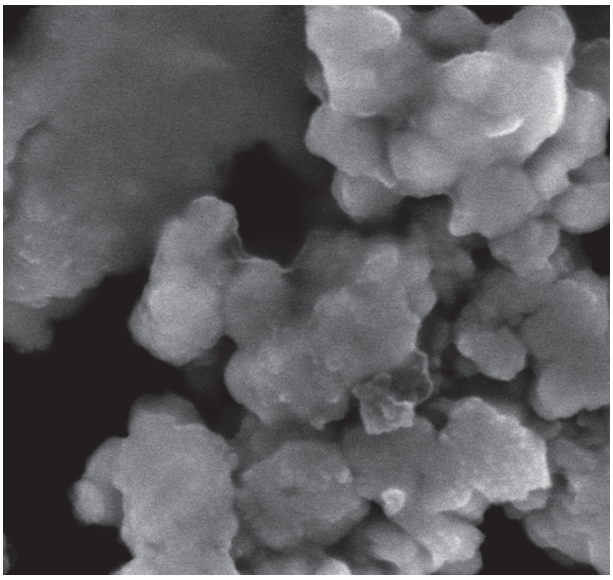

Figure 4

Figure 1 $Li_2Co_{1-X}Ni_xTi_3O_8$ and figure 2 $Li_2Co_{1-3X/2}Fe_xTi_3O_8$ present the X-ray diffractograms of the calcined pigment, at a temperature of 700°C, with a fixed time of 1 hour. The diffractograms display no intermediate phases. Figure 5 shows the UV-Vis reflectance curves versus the wavelength (nm) of the phase ($Li_2Co_{1-X}Ni_xTi_3O_8$), x=10% for temperatures of 400°C to 600°C. It can be observed that at temperatures of 400°C and 500°C, the pigments display a green-blue coloration, confirmed by the characteristic band in the 500nm region. The $Li_2CoTi_3O_8$ spinel[1] achieved stability of the green-blue colour after 600°C, figure 5. This result occurs by the modification applied during the synthesis, in which it was decided to increase viscosity, providing a greater surface area, with an increase in the dopant, and the entailing reduction in the particles, figure 6.

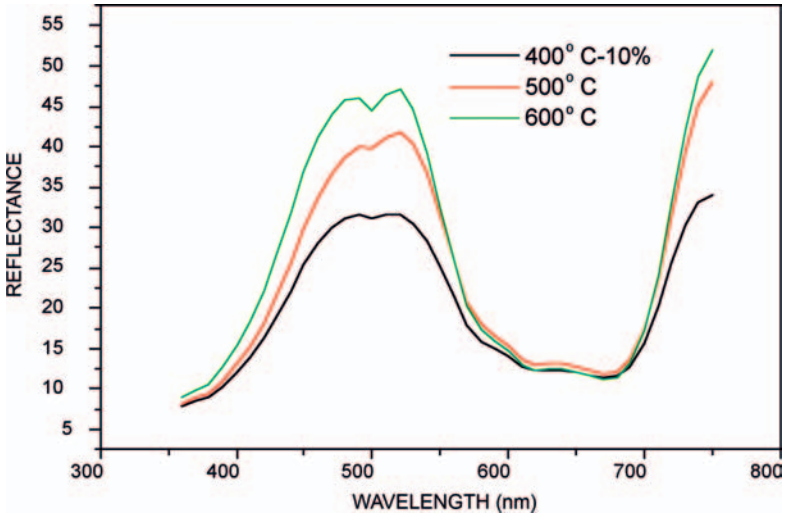

Figure 5

Figure 6

4. CONCLUSIONS

The results obtained enable drawing the following conclusions: homogeneous post crystals have been obtained, with high quality. Pigments have been produced at low temperature with high surface area by modification of the method (degree of viscosity), which provided a notable result. In accordance with the characterisation techniques used, the characteristic colour of the pigments was observed, verifying the stability of the colour with the increase in temperature and dopant. The synthesised pigments are shown to be very promising for possible application in ceramic, inks and cosmetics applications.

5. ACKNOWLEDGEMENTS

The authors are grateful for the financial support received from the Brazilian agencies CNPQ/FAPERN/LIEC.

REFERENCES

[1] M.S.C.Câmara, C.O. Paiva-Santos, E. R. Leite, E. Longo, E.J. Carda, Qualicer 2004 Pos (249-252).