MICROSTRUCTURAL AND COLOUR ANALYSIS OF $CuCrO_2$ DELAFOSSITE OBTAINED BY GELATIN METHOD

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ABSTRACT

In this paper $CuCrO_2$, the compound was synthesized by a route new that uses gelatin as organic precursor in order to application as ceramic pigments. The type of compound delafossita $CuCrO_2$ is known for its wide range of application, such as, thermoelectric devices, catalysts for the steam reforming process, ceramic pigments, NiO2 removal, among. The powders resulting from the synthesis process were calcined between 500 and 900^oC and characterized by X-ray diffraction (XRD), infrared spectroscopy, scanning electron microscopy (SEM) UV-Visible spectroscopy and Colorimetry analysis. The results showed for the $CuCrO_2$ have coloring green.

Key-words: synthesis spinel, ceramic pigments, Gelatin

1. INTRODUCTION

Ternary oxide with chemical formula $A^{+l}B^{+3}O_2$ (A=Ag, Cu, Pd e Pt; B=A1, Cr, Fe, Y, Nd, etc.). Crystallize in Delafossite structure whose name comes from the mineral Delafossite $CuFeO_2$ [1] . The structure consists of hexagonal layers formed by the ions A, B and O with compact stacking sequence [1]. The structure is highly anisotropic and can be considered as a sequence of layers of monovalent ions alternating with layers of composition BO_2 perpendicular to the c axis [1]. Oxides with delafossite structure has a variety of technological applications, such as NO_2 removal [2], thermoelectric device [3] Laser [4] catalyst [5] Field emission display (FEDs) [6] magnetic device [7,9] among others. $CuCrO_2$ delafossite is considered as p-type semiconduction for transparent electronics [4,11] and have been synthesized by self-combustion [8,10] Co-precipitation [2] and solid state reactions. However these methods require high calcinations temperature and/or long time of calcinations or aged.

This paper shows the synthesis of $CuCrO_2$ delafossite by a method that uses gelatin as an organic precursor and the microstructural and color characterization of this oxide. Gelatin is a protein derived from collagen containing various functional groups such as amino and carbonyl which have strong abilities for coordination with the metal ion. In addition to producing crystalline, nanometric and porous powders, gelatin has the advantage of being a low-cost, non-toxic material [12]. The procedure consists of forming colloidal dispersions between the gelatin and water, add metallic ions and provide heat in this dispersion for reduce volume until gel formation, which is calcined at a predetermined temperature to decompose the gelatin and to produce inorganic oxides.

2. EXPERIMENTAL

2.1 Sample Preparation

The delafossite was prepared using gelatin as organic precursor and metal nitrates as starting reagent. Initially, gelatin was added to a beaker with 150 ml of deionized water, under constant agitation, at a heating temperature of 50ºC. Cu $(NO_3)_2.3H_2O$ and Cr $(NO_3)_3.9H_2O$.were added and mixture was homogenized for 1 h. The temperature was then raised to approximately 80°C and kept under agitation until formation of a viscous gel. The gel was calcined at a temperature of 350°C for 2h at a heating rate of 10°C/min to remove the organic material. The resulting powder was calcined for 4 hours at 5°C/min at temperature of 900°C to obtain the delafossite phase.

2.2 Characterization Techniques

X-ray diffractograms were obtained with a Shimadzu XRD-6000 diffractometer, using CuKα radiation. Diffraction angles (2θ) were scanned over a range between 10° and 80º. Results were compared and analyzed with JCPDS crystallographic files. Absorption spectra in the infrared region were obtained in a Shimadzu spectrophotometer. A mixture of the sample and potassium bromide (KBr) was used in analyses. The morphology of the powders was observed in a Phillips XL-30 ESEM 20 Kv transmission electron microscope. Reflectance spectra were obtained in a SHIMADZU UV-VIS spectrophotometer, with a UV-2550 reflectance accessory, at a wavelength between 190-800 nm. A Gretag Macbeth Color-eye 2180 colorimeter was used to determine colorimetric measures of the CIEL*a*b*system. The a* coordinate varies from the red axis (a* positive) to the green (a* negative), coordinate b* from the yellow axis (b* positive) to the blue (b* negative) and L* from 0 (minimum luminosity) to 100 (maximum luminosity) on a grey scale.

3. RESULTS AND DISCUSSION

The X-ray diffraction pattern of the powder synthesized by gelatin method is shown in Fig 1. The peaks in the pattern correspond to the delafossite phase and are indexed for the space group R3m, lattice constants of a=2.856 and c=16.943 Å. in addition to the major $CuCrO_2$ phase, it is observed a peak correspond to CuO phase. In this work delafossite phase was obtained at 900 ^oC for 4 h, earlier reports have shown the preparation of $CuCrO_2$ phase in temperature above 1000 ^oC for 12 or 100 h [7].

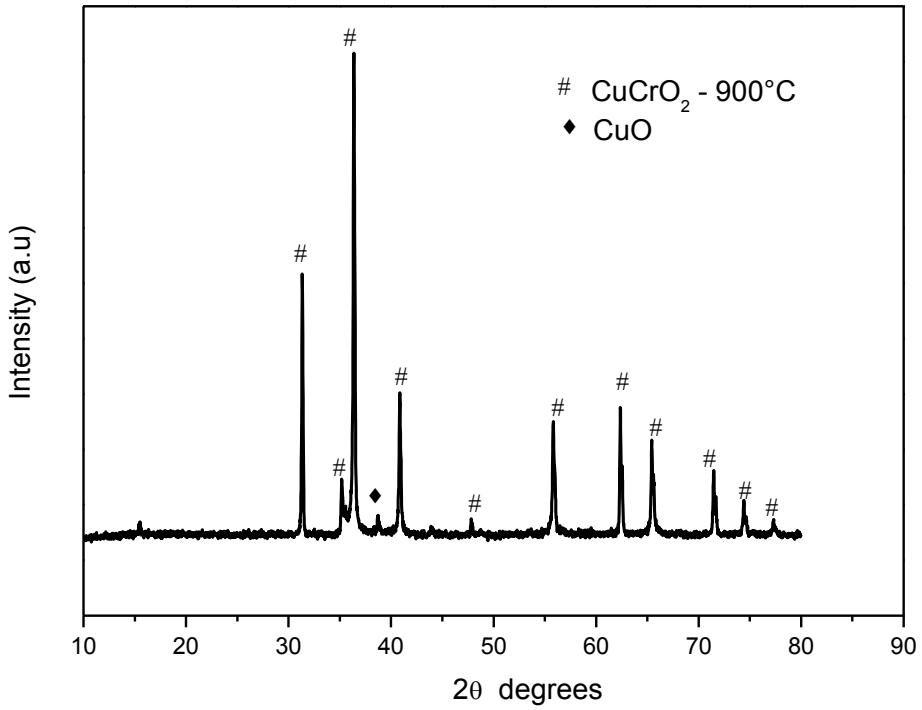

Figure 1 X-ray diffraction pattern of $CuCrO_2$.

The morphology of the delafossite $CuCrO_2$ that was prepared by gelatin method is shown in Fig 2. In this image, It is observed crystal with well-defined crystalline faces and hexagonal plates shape typical of delafossite phase.

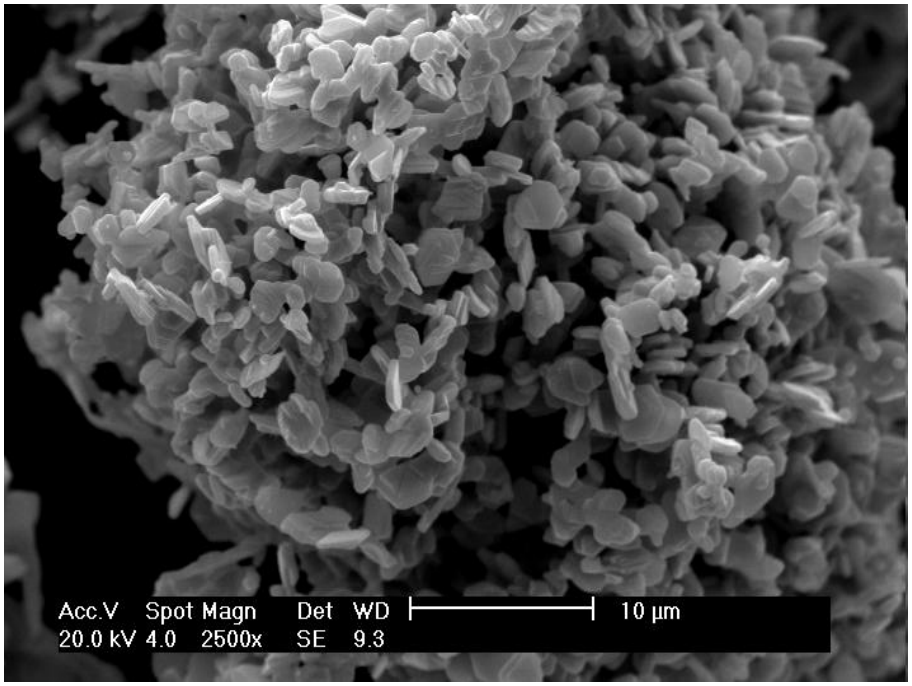

Figure 2- SEM image of $CuCrO_2$ calcined at 900°C.

Fig 3 Ilustrates the diffuse reflectance of the delafossite. We observed a band between 300 and 400 nm, low intensity region violet bands in the range of 500-550 nm, indicating that the material is absorbing in the green region. And verified low reflectance in the whole range of wavelength of visible light characteristic of low, indicating the formation of colored pigments present. [13]. This was further confirmed by the colorimetric coordinates (Table 1). The relatively high values of L* (44.96) along with low a* and b*, 2.67 and 1.24 respectively, suggested a green pigment.

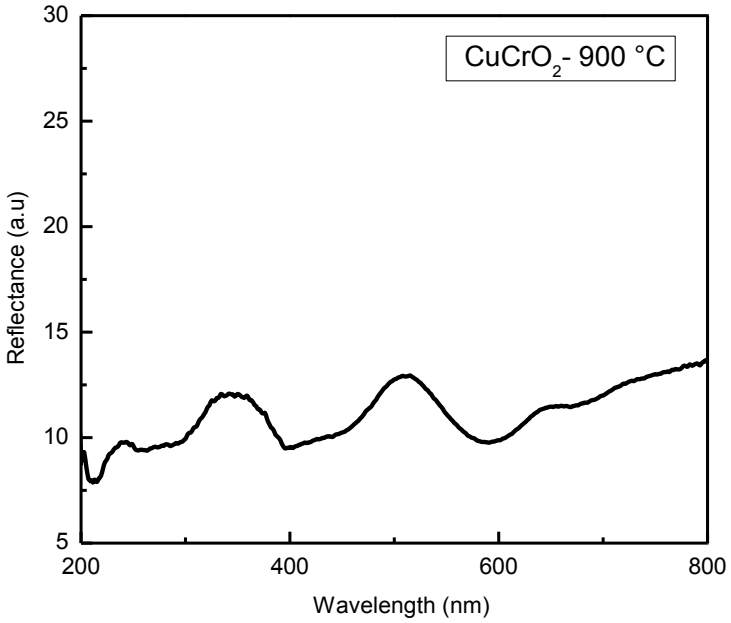

Figure 3 Diffuse reflectance of pigment $CuCrO_2$.

Tabela 1: Colorimetric coordinates of $CuCrO_2$

AMOSTRA	T(C°)	L*	a*	b*	CORES
$CuCrO_2$	900	44,96	-2.67	1.24	green

4. CONCLUSIONS

Results indicate that synthesis using gelatin as organic precursor is a favorable process to obtain single phase delafossite structure at 900 ^{o}C. Curvas de reflectância e coordenadas cromáticas revelou que os pigmento delafossite ($CuCrO_2$), apresentou coloração verde.

ACKNOWLEDGEMENTS

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