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Characterization of commercial luminescent powders

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Abstract. Luminescent powders have attracted the attention of scientists, boosting properties as specific as mechanoluminescence, thermoluminescence, and photoluminescence among other characteristics. The uses of these properties have grown exponentially from recreational uses in luminescent paints, toys, plasticines, and other industry-specific uses, such as crack sensors, flaw detectors, and radiation meters in medicine. These uses have grown proportionately with their more economical and efficient manufacturing methods and processes, generating more economical fluorescent pigments. This paper characterizes and compares a low-cost luminescent pigment with those reported by different authors, its chemical composition was characterized by X-ray energy dispersion spectrometry, X-ray fluorescence, and Raman spectroscopy. Its morphology was analyzed by scanning electron microscopy, and its particle size with a laser meter its structural condition with X-ray diffraction, the powder obtained was presented as a strontium aluminate doped with divalent europium and trivalent dysprosium (Al₂O₄: Eu²⁺, Dy³⁺) one of the most efficient powders around persistence and luminance intensity at present. The structure of the strontium aluminate was determined by the main peaks of the diffractogram showing a monoclinic system. The elemental composition helped to determine the doping of the strontium aluminate corroborating them with the obtained with Raman spectroscopy and the scanning electron microscopy images.

1. Introduction

Luminous powders are the basis for the manufacture of millions of products in the world, these powders have evolved reaching a persistence of up to 24 hours and much higher emission intensity. These improvements have occurred, adding doping with ions of different elements such as rare earth, which allows these emission matches to improve their quality and surprise the world with their applications.

The most common luminescent powders are strontium aluminates, which were doped with divalent europium for the first time in 1968 [1] with an emission peak of 520 nm and producing ultraviolet excitation in the host network of the electronic configuration 4f⁷ from divalent europium to excited states 4f⁷ and 4f⁶ and 5d¹ of the same [2]. In 1996 the trivalent ion of dysprosium was included Dy⁺³, causing a more intense phosphorescence and converting from this moment the strontium aluminate doped with europium and dysprosium into the persistent luminescence powder par excellence [3].

These powders have been used for fluorescent lamps [1], luminous paints [4], types of ecological cement [5] and light intensity sensors [6], as well as being the most promising materials for structural health monitoring applications [7]. In 2014 these compounds were used to manufacture films with epoxy resins with a ratio of 3:1 generating a phenomenological model of tension-optical transduction to predict mechano-luminescence [8], from SrAl₂O₄:Eu²⁺ compounds that are known to have better behavior for



dynamic visualization of cracks due to their high sensitivity, and also SrAl₂O₄:Eu²⁺, Dy³⁺ that is suitable for the complete visualization of deformations due to their better linearity.

In 2008 the triboluminescent properties of SrAl₂O₄ were studied: Eu²⁺ on a 600 Inconel steel deposited by radiofrequency spraying showing a preferential orientation in (0 3 1) in addition to presenting luminescence to stress [9] proving its mechano-luminescence as one of the most promising uses of these powders.

Among the most common manufacturing processes to obtain doped strontium, aluminates are solid-state synthesis [10], synthesis by molten salts in which different compounds act to regulate the size of the coexisting monoclinic phase of crystals at low temperatures and hexagonal at high temperatures. The synthesis by combustion produces in these materials laminar morphology with low temperatures of manufacture and high crystallinity, these processes of manufacture have been optimized diminishing the costs of manufacture and generating some uses in the industry and more specifically in the Colombian one which has not deepened the use of these powders.

2. Materials and methods

The low-cost commercial 3D fluorescent pigment powders were characterized by the Mastersizer 3000 laser diffraction particle size analyzer in a water dispersing medium. The morphological and elemental characterization of the powders was performed by scanning electron microscopy / energy dispersive X-ray spectroscopy (SEM/EDS) using a Zeis Evo MA10.

The chemical and structural composition of the commercial powders was determined by X-ray fluorescence (XRF) in the brand PANalytical MiniPal 2, the structural characteristics were studied through the X-ray diffractometer, PANalytical X'Pert PRO PW500 with cobalt cathode was scanned at angles between 25° and 50° for 15 minutes. In the study of the composition by Raman spectroscopy, the sample was executed in powder form in the brand thermoscientific equipment.

3. Results and discussion

The particle size of the powders varies from 20 μm to 45 μm as shown in Table 1 and has a specific average surface of 195.6 m²/kg that coincides with the powders obtained at an industrial level through solid-state synthesis. This process handles treatment temperatures of up to 1900 °C and long processing times to increase its specific surface in luminescence [11].

Table 1. Particle size.

size (μm)	% Volume below
20	8.27
25	12.47
32	19.44
38	25,85
45	33.20

In the micrographs present in Figure 1 taken at 5000 X, bulk grains in the matrix and small adhering crystals are observed which will result in an increase in grain size due to coalescence phenomena. Figure 1 shows surface 2-B with particle sizes coinciding with Table 1, with an elemental composition by X-ray scattering spectrometry dominated by aluminum, strontium, and dysprosium as shown in Table 2. In Figure 2 in point 2-C to 5000 X, we can see the morphology of the particle in which reference is made to the predominant matrix, which was characterized by x-ray dispersion spectrometry obtaining an aluminum and strontium composition as shown in Table 2. In point 2-A of Figure 2, adhesions to the grain are observed, which indicate the composition of the doping reported in Table 2, finding a representative increase of dysprosium in addition to the europium.

Table 3 reports X-ray fluorescence corroborating the components found by X-ray scattering spectrometry, where the same components of Table 2 were detected with a more representative content of europium and dysprosium, main components of the most common luminescent powder strontium

aluminate doped with divalent ions of europium and trivalent of dysprosium. titanium is possibly derived from the manufacturing process of superimposing its assay energy.

In X-ray diffraction, we observe very refined peaks, which shows us an element with a large crystal size, where the SrAl_2O_4 phase prevails dominated by a monoclinic system with unit cell parameters $a = 8.4470 \text{ \AA}$, $b = 8.8160 \text{ \AA}$, $c = 5.1630 \text{ \AA}$ $\beta = 93.420^\circ$ and a density of 3.557 g/cm^3 . Where the representative peaks with orientation (0 3 1) Figure 3 peak 4 are reported by Fu [9] these peaks are characteristic of SrAl_2O_4 reported by [12] with orientations in their main peaks Figure 3, (2 1 1) peak 1, (2 2 0) peak 2, (2 1 1) peak 3, (0 3 1) peak 4.

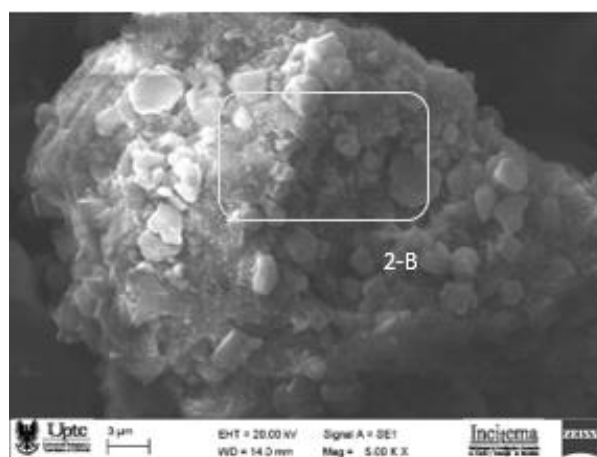


Figure 1. Scanning electron microscopy bulk luminescent powders.

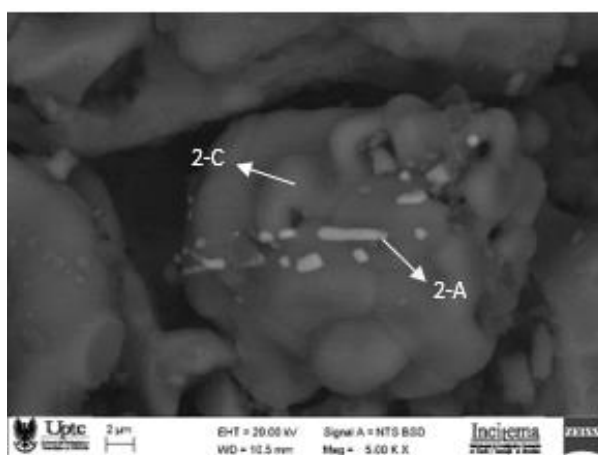


Figure 2. X-ray scattering spectrometry spots.

Table 2. X-ray scattering spectrometry luminescent powders.

Element	2-B Weight%	2-A Weight%	2-C Weight%
O K	51.89	31.54	38.01
Al K	21.51	15.57	22.93
Sr L	26.41	15.49	39.07
Dy L	0.20	35.45	----
Eu L	----	1.94	---
Totals	100.00	100.00	100.00

Table 3. X-ray fluorescence of luminescent powders.

Al	Sr	Eu	Dy	Ti
22.5%	76.4%	0.40%	0.34%	0.17%

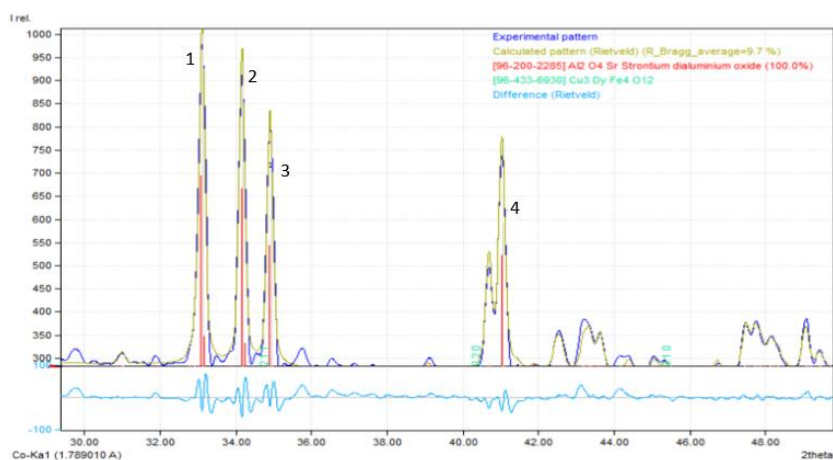


Figure 3. X-ray diffraction of luminescent powders.

The Raman spectrum of the powders can be found in Figure 4, where the signals from 0 to 3500 cm^{-1} are evident, the representative peaks of SrAl_2O_4 are shown. Figure 5 shows a close-up of the spectrum from 0 cm^{-1} to 1500 cm^{-1} , in which we find that the peak that is approximately 465 cm^{-1} corresponds to the torsional vibration of the O-Al-O link [11] in addition to the less intense vibration mode found in 795 cm^{-1} the two states related to SrAl_2O_4 .

The presence of Raman peaks located approximately at 328 cm^{-1} corresponds to the Eu-O tension vibration, the peaks attributed to Eu doping are located at 117 cm^{-1} , 144 cm^{-1} , 284 cm^{-1} , 340 cm^{-1} , 383 cm^{-1} of the dysprosium doping peak may be overlapped by the O-Al-O link peak and the Eu peak as its most representative peaks are at 370 cm^{-1} , 328 cm^{-1} , 462 cm^{-1} , 1100 cm^{-1} [11].

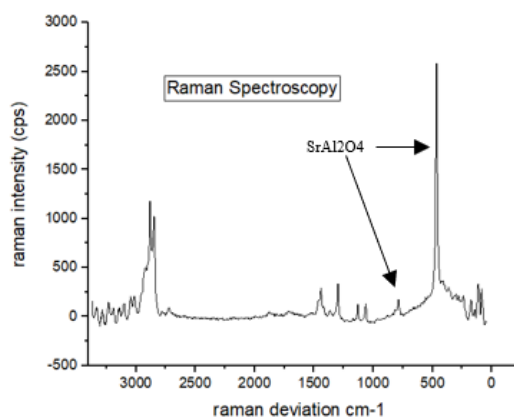


Figure 4. Raman spectrum from 0 cm^{-1} to 3500 cm^{-1} .

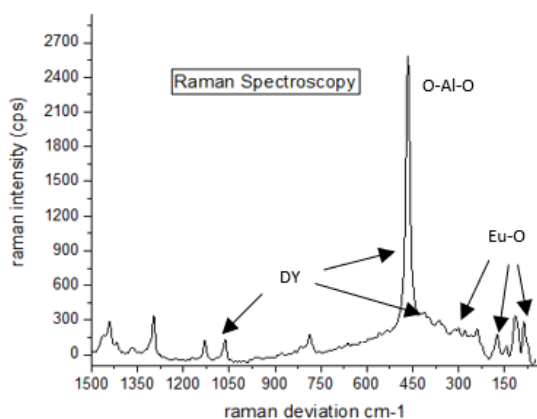


Figure 5. Raman spectrum from 0 cm^{-1} to 1500 cm^{-1} .

4. Conclusions

It was found that low cost 3D luminescent powders are strontium aluminates doped with europium and dysprosium, doping was detected by scanning electron microscopy and subjected to scattering spectroscopy these points which demonstrated the presence of europium and dysprosium in the adhered crystals and strontium aluminate in the matrix.

With x-ray diffraction, the main strontium aluminate peaks were found that match those found in the literature, it could be observed by the shape of the peaks which is common in compounds with high crystallinity. The main vibrational states of the strontium aluminate bonds doped with europium and dysprosium are reported. These powders can be an innovation option in many products and applications for companies in Colombia.

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