

Rare-Earth Doped GdVO₄ by Sol-Gel Method: Structural and Luminescence Properties

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Abstract

Rare earths have been extensively developed in recent years, however, new hosts allow high excitation and emission efficiency, in this sense, gadolinium vanadate has been extensively studied and in previous works it has been widely used in down conversion systems. Because of the strong absorption of the VO₄ groups and efficient energy transfer from GdVO₄ to lanthanide ions, in this work its up-conversion properties were studied when is co-doped with Yb³⁺, X³⁺ where X = Tm, Er and Ho. The powders synthesized presented a high crystallinity and a rounded morphology and exhibit a high luminescence when are excited with IR radiation.

Keywords

Gadolinium, Rare Earths, Up Conversion

1. Introduction

Rare earths (RE) are widely studied due to their unique properties and applications in optoelectronic, antibacterial activity, and other fields [1].

LnVO₄ orthovanates (Ln = Y, Gd, ..., Er) have been used for many years as optical materials: phosphors, luminescent probes, fiber laser hosts, etc. [2]. It has been observed that in orthovanadate crystal lattices, the excitation of rare earth

ions takes place mainly due to the absorption of excitation light by VO_4^{3-} vanadate molecular groups [3].

Gadolinium vanadate (GdVO_4) is a material with diverse applications as a nanoscopic material combining magnetic resonance contrast enhancement properties with luminescence properties and hydrogen peroxide sensing characteristics [4], donor in fluorescence resonance energy transfer (FRET) in aqueous solution [5] and for multimodal imaging functionalities and its performance as a radionuclide carrier for targeted alpha therapy [6].

For the equal valence and similar ionic radii the Gd compounds can be doped with luminescent lanthanide ions. They can be efficiently excited with UV radiation because of the strong absorption of the VO_4 groups and efficient energy transfer from GdVO_4 to lanthanide ions. Therefore, GdVO_4 is used as a phosphor (doped with Eu^{3+} , Dy^{3+} , Sm^{3+}), an upconverter (doped with $\text{Er}^{3+}/\text{Yb}^{3+}$, $\text{Ho}^{3+}/\text{Yb}^{3+}$, or $\text{Tm}^{3+}/\text{Yb}^{3+}$) and a laser (doped with Nd^{3+}).

Several methods have been used to prepare undoped and RE-doped GdVO_4 . $\text{GdVO}_4:\text{Eu}^{3+}$ have been successfully synthesized via a facile solvothermal route, a urea hydrolysis method, a co-precipitation synthesis, as well as facile hydrothermal methods.

$\text{Ho}^{3+}/\text{Yb}^{3+}$ co-doped GdVO_4 nanophosphors have been synthesized via a facile modified sol-gel Pechini method, these samples were annealed at 900°C for 4 h.

In the present work we show the results obtained to carry out the synthesis of luminescent powders of $\text{GdVO}_4:\text{Yb}^{3+}$ (Itrbium), X^{3+} [$\text{X} = \text{Tm}$ (Thulium), Er (Erbium) and Ho (Holmium)].

As previously mentioned, rare earths are a group of elements that constitute a set of substances of interest with optoelectronic applications, that is, applications in the field of telecommunications.

The need of human beings to keep in constant and fast communication has led to the creation of different means to perform this action with good efficiency, from the sending of pigeons with messages to the creation of optical fiber.

The main function of optical fiber is to transmit light signals at different frequencies at high speed, thanks to the fact that it has a much lower degree of attenuation and interference. However, there are studies that show that at a certain distance the frequency of the transmitted light suffers a decrease in the signal, causing the message to take much longer to reach its destination, so it is necessary to solve this problem, and glasses doped with rare earths have shown satisfactory results.

2. Methodology

2.1. Materials

Gd_2O_3 (Gadolinium oxide, Sigma Aldrich, 99.9%), Itrbium nitrate ($\text{Yb}[\text{NO}_3]_3$, Sigma-Aldrich, 99.999%) and thulium oxide (Tm_2O_3 , Sigma-Aldrich, 99.9%) were used as precursors. While ammonium metavanadate (NH_4VO_3 , Sigma Al-

drich, 99.9%) as well as erbium oxide (Er_2O_3 , Sigma Aldrich, 99.99%) and holmium chloride (HoCl_3 , Sigma Aldrich, 99.9%) were used as matrix for the doping agents.

2.2. Equipment

- *FTIR (Fourier transform infrared spectroscopy) and XRD (X-ray diffraction).*

Infrared spectroscopy analysis by FTIR was performed using a Shimadzu IR-Tracer-100 spectrophotometer. The FTIR spectra were recorded in the energy range $400 - 4000 \text{ cm}^{-1}$ and the attenuated total reflection (ATR) accessory. Attenuated total reflection (ATR).

- *Photoluminescence characterization.*

Spectra were obtained using a Princeton Instruments Acton Spectra Pro modular fluorometer equipped with a 75 W xenon lamp (Newport Oriel). It also has excitation (SP2300i) and emission (SP-2500i with a Hamamatsu R955 photomultiplier attached) monochromators.

- *Structural and morphological characterization.*

The diffractograms were obtained using a Bruker D8 Advance X-ray diffraction Bruker D8 Advance X-ray diffraction (XRD) equipment emitting Cu-K α radiation (wavelength 1.5418 \AA). XRD patterns were recorded in the 2θ range of $20^\circ - 70^\circ$ at a scan rate of $0.1^\circ/\text{s}$.

Scanning electron microscopy scanning electron microscopy (SEM) was obtained with the FEI-ESEM QUANTA 250 equipment to observe the morphology of the glasses.

Finally, transmission electron microscopy (TEM) was used with a FEI-Titan 80 - 300 microscope and a power of 12 KV.

2.3. Preparation

The first step to obtain the metal salts was to convert the oxides to nitrates using nitric acid (HNO_3 , Sigma Aldrich, 70%) with a 1:1 volume ratio according to Kolesnikov [7]. The dissolved oxides were subjected to stirring for a time of 1 h at a temperature of 80°C . When the solutions were still transparent, the resulting solution was allowed to cool to room temperature and then subjected to a drying process at a temperature of 100°C for 24 h. Finally, to obtain gadolinium and erbium powders, heat treatment at a temperature of 200°C for 20 minutes was necessary. The X-ray diffraction technique was used to corroborate the structure of the nitrates.

After the oxide was converted to nitrates, gadolinium nitrate at 1M, ytterbium nitrate at 3 atomic percent and holmium chloride at X atomic percent ($X = 1, 3, 5$) were dissolved in ethanol-water solution at 113 M with a ratio of 1:1 by stirring at a temperature of 60°C for a time of 30 minutes. After that time, citric acid at 2.6 M as a chelating agent, ammonium metavanadate at 1.3 M as a precursor and ethylene glycol at 0.37 M as a stabilizer was added with constant stirring at a temperature of 80°C for 1 h and at 800 rpm.

The sol was kept under stirring until the solution turned dark blue in color. The resulting solution was dried at a temperature of 100°C for 24 h to remove excess water and alcohol. To promote crystallization of the powders, a thermal treatment (T.T.) was necessary at temperatures of 600°C, 700°C, 800°C, 900°C and 1000°C for a time of 1 h. **Table 1** shows the stoichiometric quantities of the elements used in the matrix with Yb and **Table 2** enlisted the amounts in percentage of each of rare earths used in the experiment.

3. Results and Discussion

Formation, morphology and structure analysis.

3.1. FTIR and XRD

Once the coincidence of the crystalline phase of $GdVO_4$ with the 17-0260 card was corroborated, X-ray diffraction was performed to obtain the diffractograms of each of the resulting powders of $GdVO_4:Yb^{3+}$, X^{3+} ($X = Tm, Ho, Er$) with heat

Table 1. Stoichiometric quantities of the elements used in the matrix with Yb.

Solvent		Rare-earth ions	
Ratio	1:1	Gd (NO ₃) ₃	0.343 g
Ethanol	5 ml	NHVO ₃	0.116 g
H ₂ O	5 ml	Yb (NO ₃) ₃ *	0.022 g
0.1 Molar		*3% atm	
Chelating agent			
g citric acid		0.384	
Modifier			
ml ethylene glycol		0.027	

Table 2. Synthesized systems of the Tm, Er, and Ho ions at different TT.

Dopant System: GdVO ₄		
Yb	3%	0.022 g
Tm		
Er	1%	0.007 g
Ho		
Tm		
Er	3%	0.022 g
Ho		
Tm		
Er	5%	0.038 g
Ho		

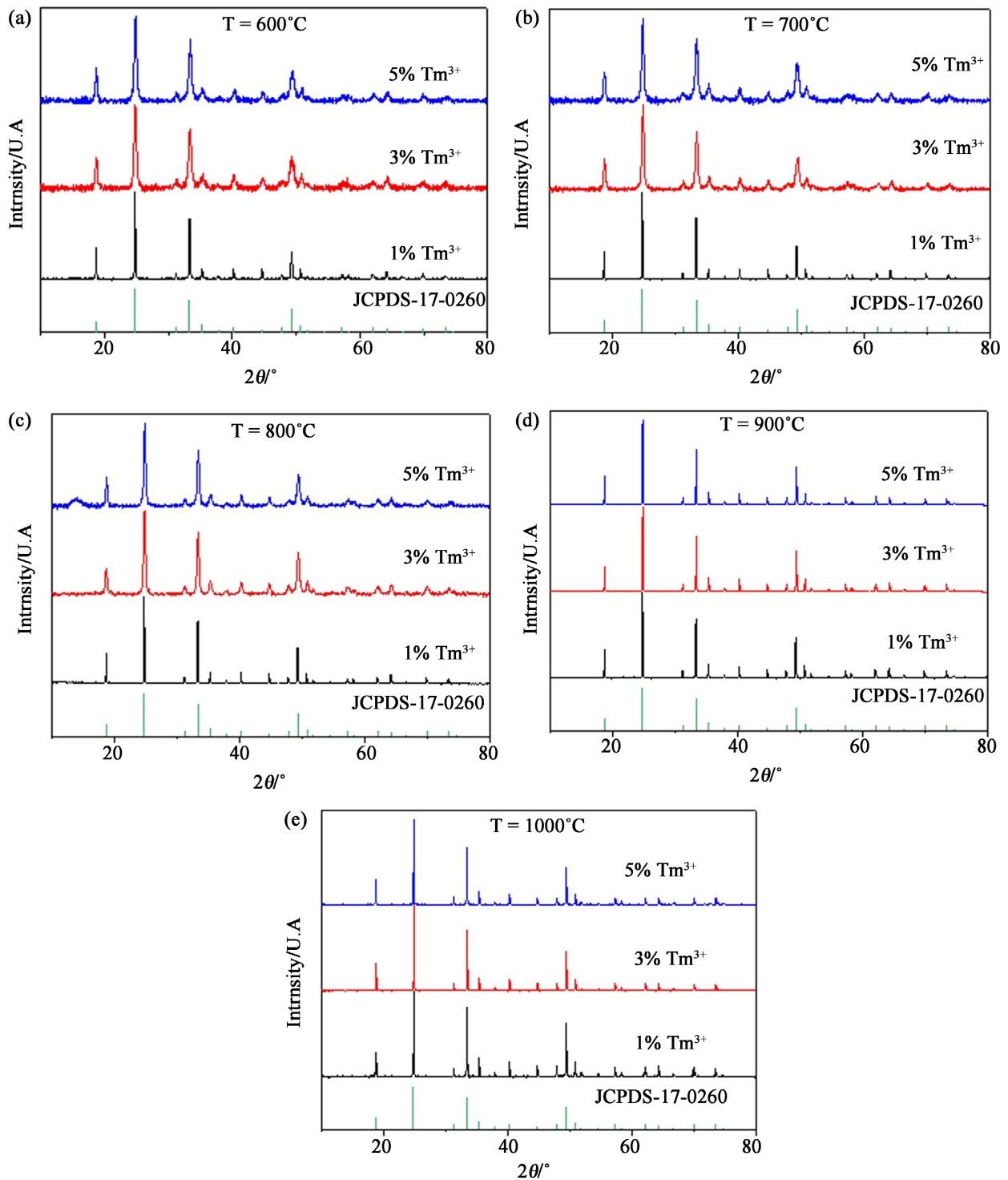


Figure 1. Diffractogram of the GdVO_4 system: Yb^{3+} , Tm^{3+} (1%, 3%, and 5% at.) at (a) 600 °C, (b) 700 °C, (c) 800 °C, (d) 900 °C, and (e) 1000 °C.

treatment of 600 °C, 700 °C, 800 °C, 900 °C and 1000 °C.

Figure 1 shows diffractograms of the system $\text{GdVO}_4:\text{Yb}^{3+}$, Tm^{3+} (1%, 3%, and 5% at.) at (a) 600 °C, (b) 700 °C, (c) 800 °C, (d) 900 °C, and (e) 1000 °C. From the results obtained, it is evident that the crystallite size is larger at 1% atm of Tm^{3+} up

to 800°C, once this temperature is exceeded, the crystallite size is larger at 3% atm of Tm^{3+} ; while the smallest crystallite was found to be in most cases 5% at. of Tm^{3+} .

Figure 2 shows diffractograms of the system $\text{GdVO}_4:\text{Yb}^{3+}$, Er^{3+} (1%, 3%, and 5% at.) at (a) 600°C, (b) 700°C, (c) 800°C, (d) 900°C, and (e) 1000°C. From the results obtained, it is evident that the crystallite size is larger at 1% at. of Tm^{3+} up to 800°C, once this temperature is exceeded, the crystallite size is larger at 3% at.

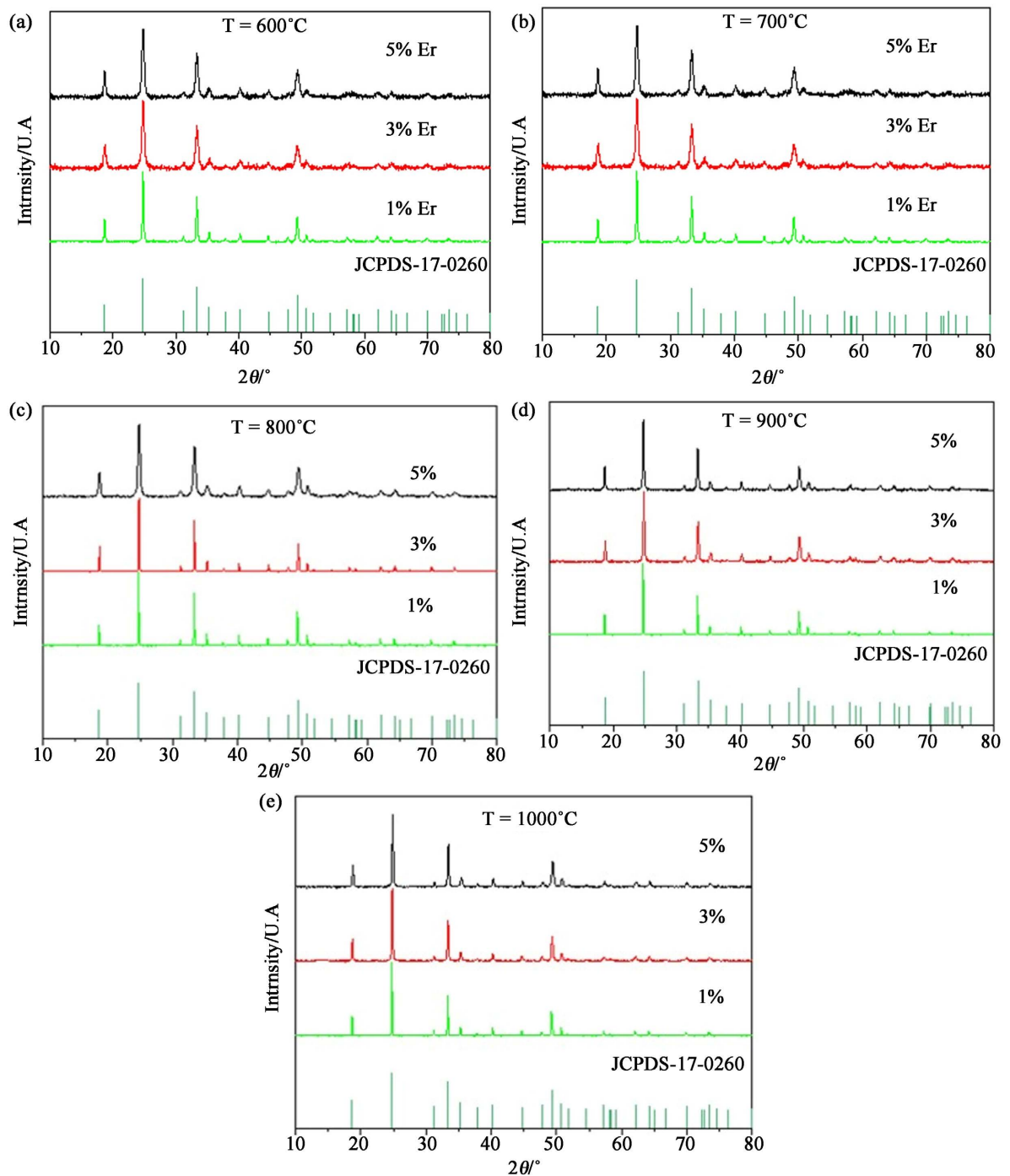


Figure 2. Diffractogram of the GdVO_4 system: Yb^{3+} , Er^{3+} (1, 3, and 5% at.) at (a) 600°C, (b) 700°C, (c) 800°C, (d) 900°C, and (e) 1000°C.

of Tm^{3+} ; while the smallest crystallite was found to be in most cases 5% at. of Tm^{3+} . According to the results obtained, it is noticeable that the largest crystallite size is with 1% at. of Er^{3+} ; while the smallest crystallite turned out to be in most cases 5% at. of Er^{3+} .

Figure 3 shows diffractograms of the system $\text{GdVO}_4:\text{Yb}^{3+}, \text{Ho}^{3+}$ (1%, 3%, and 5% at.) at (a) 600°C, (b) 700°C, (c) 800°C, (d) 900°C, and (e) 1000°C. According to

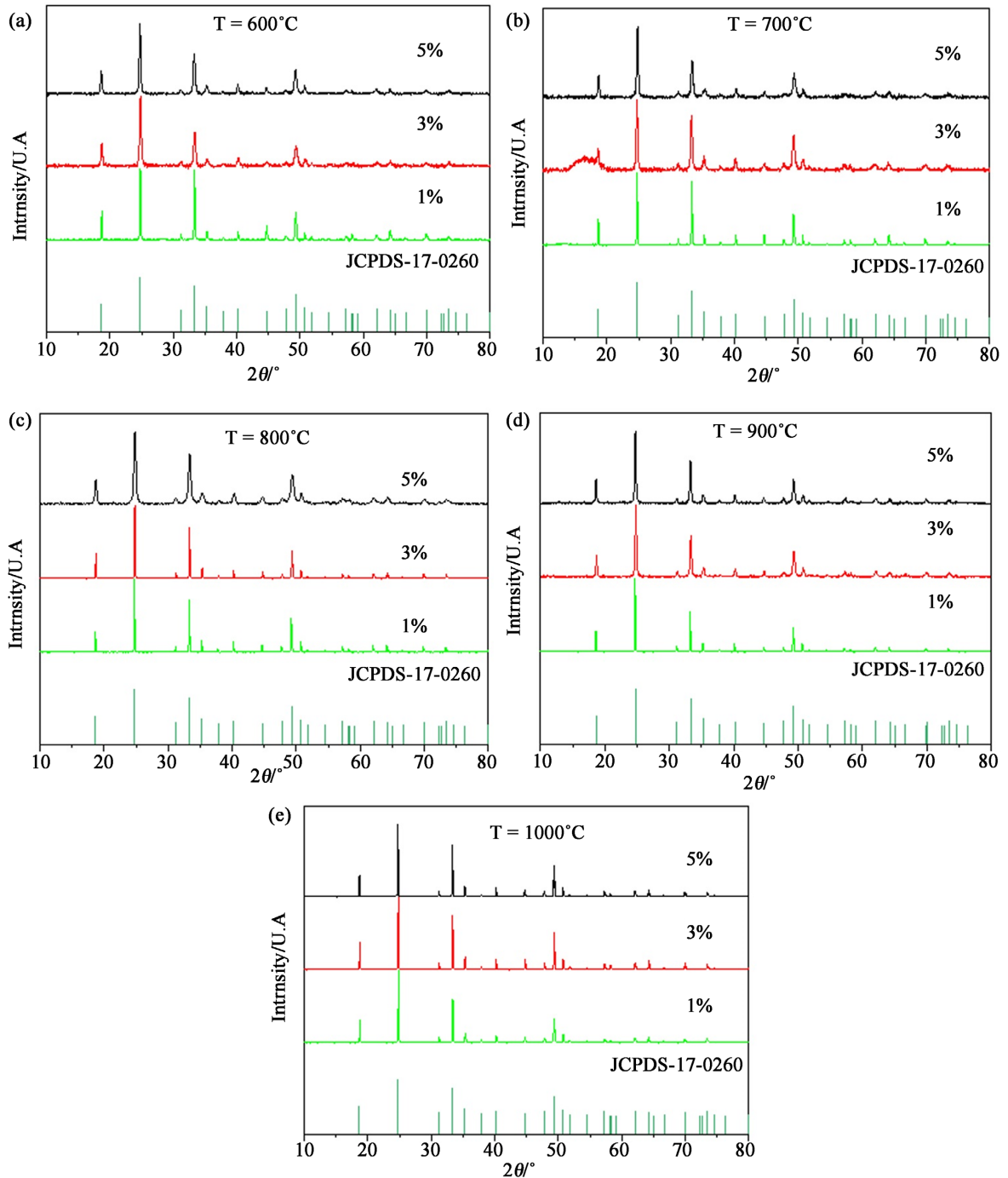


Figure 3. Diffractogram of the GdVO_4 system: $\text{Yb}^{3+}, \text{Ho}^{3+}$ (1%, 3%, and 5% at.) at (a) 600°C, (b) 700°C, (c) 800°C, (d) 900°C, and (e) 1000°C.

the results obtained, it is evident that the largest crystallite size belongs mostly to 1% at. of Ho^{3+} ; while the smallest crystallite turned out to be mostly 5% at. of Ho^{3+} .

Finally the **Table 3** shown the Diffractogram of the GdVO_4 system: Yb^{3+} , Ho^{3+} (1%, 3%, and 5% at.) at (a) 600°C, (b) 700°C, (c) 800°C, (d) 900°C, and (e) 1000°C.

In where is shown that when the temperature increase the crystalline size also increased, for all the system presented

3.2. Photoluminescence Study

3.2.1. GdVO_4 Luminescence Spectrometry of Yb^{3+} and Tm^{3+} at Different Temperatures

Figure 4 shows GdVO_4 luminescence spectrometry of Yb^{3+} and Tm^{3+} at 800°C. It

Table 3. Diffractogram of the GdVO_4 system: Yb^{3+} , Ho^{3+} (1%, 3%, and 5% at.) at (a) 600°C, (b) 700°C, (c) 800°C, (d) 900°C, and (e) 1000°C.

T.T./°C	Crystalline size/nm		
	Tm (3%)	Er (3%)	Ho (3%)
600	10.38	13.78	18.18
700	11.31	19.68	19.09
800	13.89	25.26	25.91
900	38.17	22.58	36.51
1000	38.33	24.11	32.77

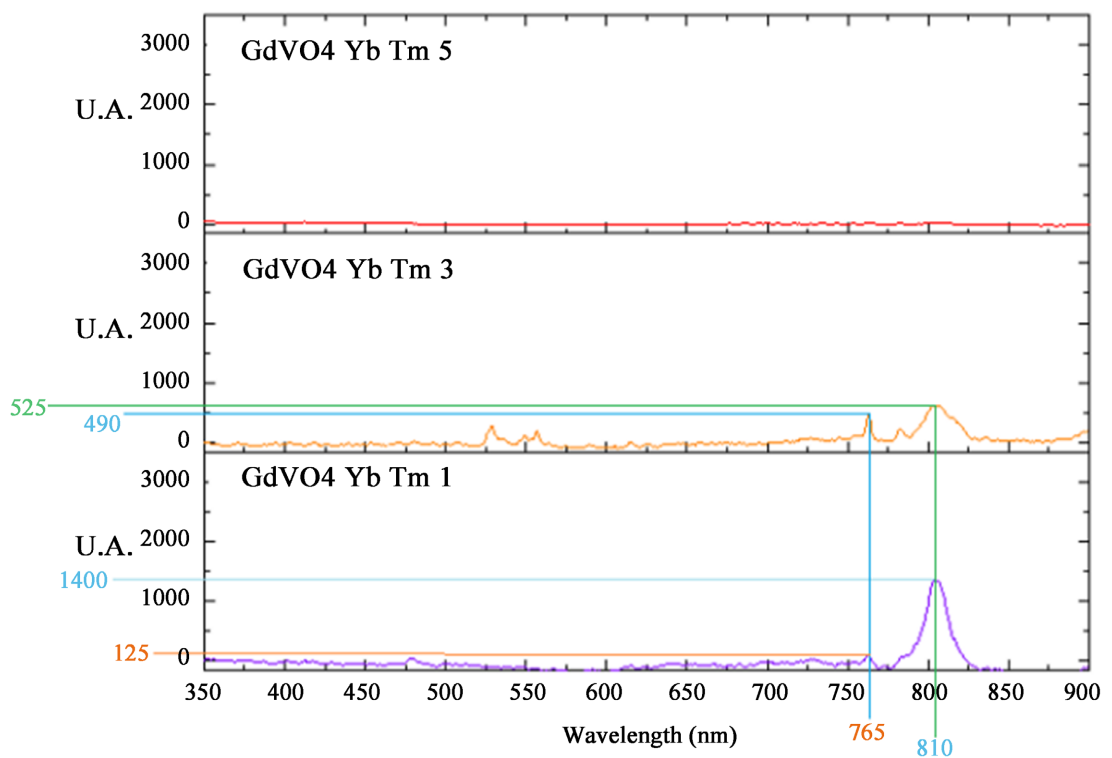


Figure 4. GdVO_4 luminescence spectrometry of Yb^{3+} and Tm^{3+} at 800°C.

observes that the wavelength (X-axis) for both 3% and 1% of the maximum and minimum points are exactly the same for these percentages. But its absorbance does show changes, for 3% it has a maximum point of 525 AU and a minimum of 490 AU; while for 1% it has a maximum of 1400 AU and a minimum of 125 AU.

As a result, the highest spectrometry point belongs to 1% with values of 810 nm and 1400 AU.

- *GdVO₄ luminescence spectrometry of Yb³⁺ and Tm³⁺ at 1000 °C.*

Considering **Figure 5** and the data in **Table 4**, it is evident that for the 5% crystal at T = 1000 °C its luminescence just begins, with values of 806 nm and 300 AU; on the other hand, the crystal with 3% has a slightly larger wavelength (0.25 nm) compared to the other 2 percentages.

The crystal with 1% presents the largest luminescence peak, with values of 806 nm - 900 AU, for wavelength and absorbance respectively.

Finally concluding that luminescence spectrometry of GdVO₄ from ytterbium

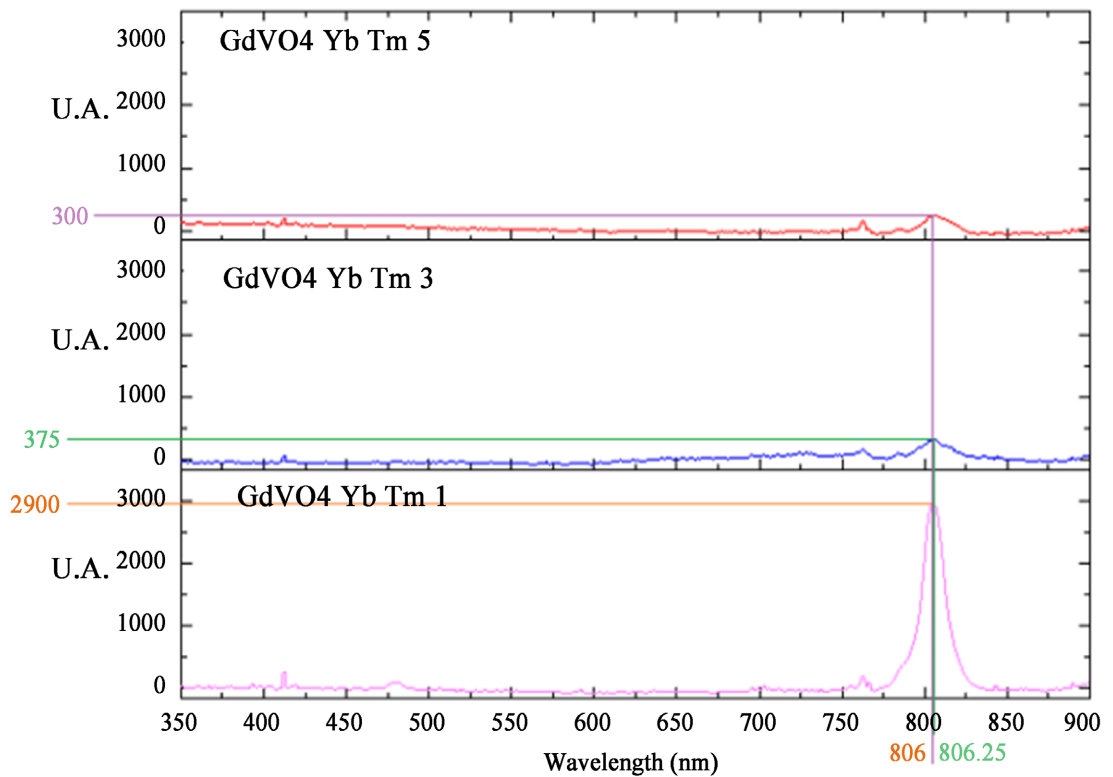


Figure 5. GdVO₄ luminescence spectrometry of Yb³⁺ and Tm³⁺ at 1000 °C.

Table 4. Wavelength and absorption of the highest GdVO₄ spots of Yb³⁺ and Tm³⁺ at 1000 °C.

Crystallite size	Wavelength (nm)	
	x	y
5%	806	300
3%	806.25	375
1%	806	900

(Yb³⁺) and thulium (Tm³⁺) has better results when the temperature is elevated, and the 1% crystal has the best luminescence peaks in both cases (T = 800°C and T = 1000°C).

3.2.2. GdVO₄ Luminescence Spectrometry of Yb³⁺ and Ho³⁺ at Different Temperatures.

- *GdVO₄ luminescence spectrometry of Yb³⁺ and Ho³⁺ at 800 °C. According to Figure 6 and Table 5, it was obtained:*

*For the 5% crystal, its highest values were 803 nm and 510 AU.

*For the 3% crystal, its highest values were 807 nm and 760 AU.

*For the 1% crystal, its highest value for wavelength was 665 nm (corresponding to an absorbance of 1950); while its highest absorbance value was 2200 (corresponding to a wavelength of 653 nm).

Therefore, it is determined that the 1% crystal has better results in both wavelength and absorbance.

- *GdVO₄ luminescence spectrometry of Yb³⁺ and Ho³⁺ at 1000 °C.*

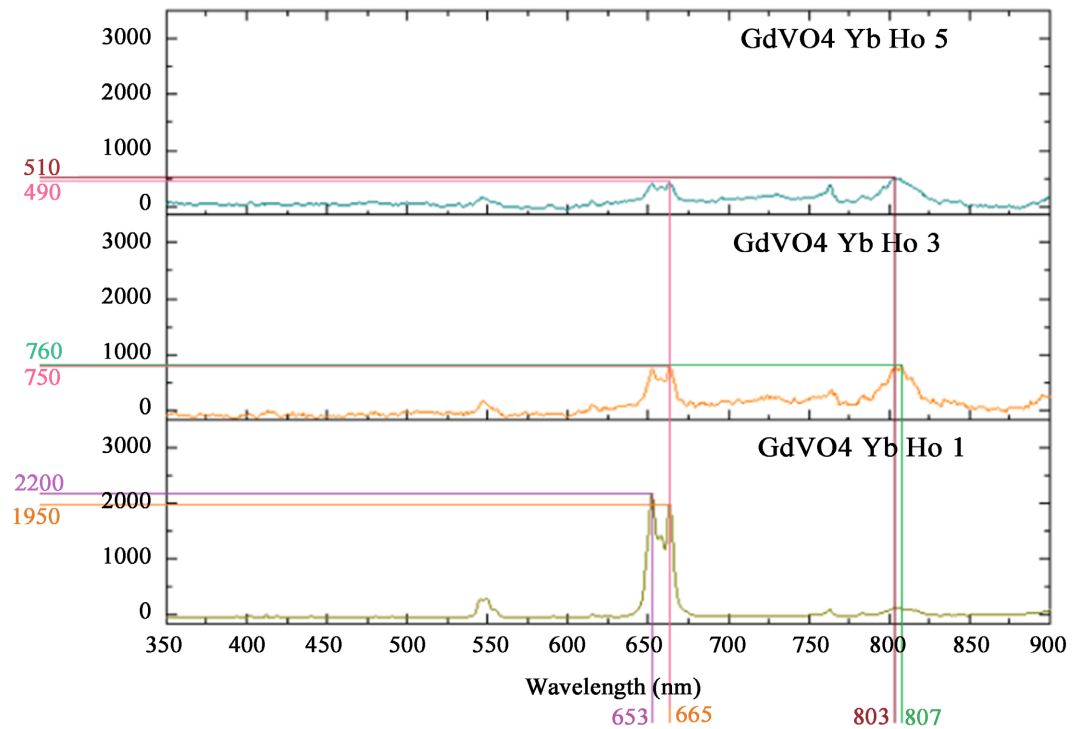


Figure 6. GdVO₄ luminescence spectrometry of Yb³⁺ and Ho³⁺ at 800 °C.

Table 5. Wavelength and absorption of the highest GdVO₄ spots of Yb³⁺ and Ho³⁺ at 800 °C.

Crystallite size	Wavelength (nm)		Absorption (U.A)	
	x1	x2	y1	y2
5%	803	665	510	490
3%	807	665	760	750
1%	653	665	2200	1950

Figure 7 and **Table 6** show that the lowest luminescence was presented by the 3% crystal, with an absorbance of 490 AU; while the 1% crystal has the highest absorbance with 3150 AU. As for the wavelength values for the 1%, 3% and 5% crystals, they all present their peaks at the same values, 665 nm for the highest peak and 652 nm for the second highest peak. The 1% crystal presents the largest luminescence peak, with values of 665 nm - 3150 AU, for wavelength and absorbance respectively.

It is concluded that GdVO₄ luminescence spectrometry of Ytterbium (Yb³⁺) and Holmium (Ho³⁺) has better results when the temperature is elevated, and the 1% crystal has the best luminescence peaks in both cases (T = 800°C and T = 1000°C).

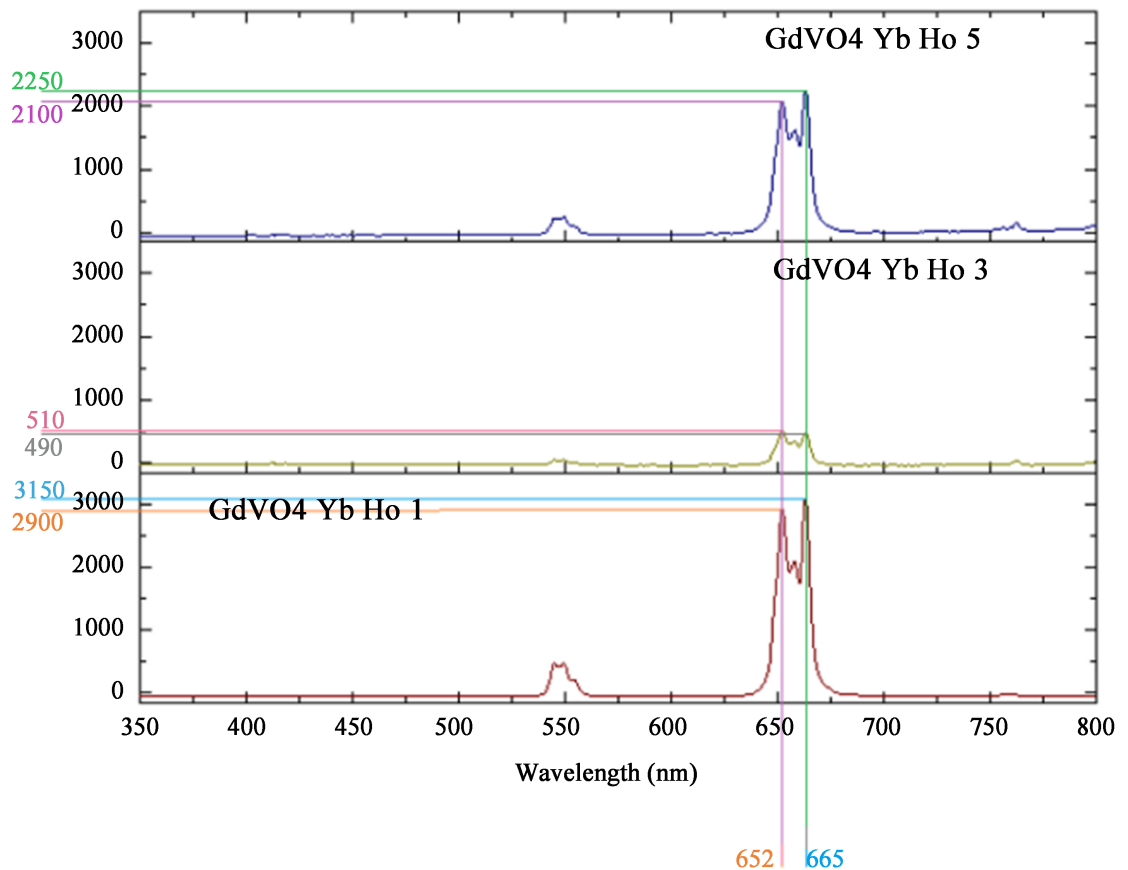


Figure 7. GdVO₄ luminescence spectrometry of Yb³⁺ and Ho³⁺ at 1000°C.

Table 6. Wavelength and absorption of the highest GdVO₄ spots of Yb³⁺ and Ho³⁺ at 1000°C.

Crystallite size	Wavelength (nm)		Absorption (U.A)	
	x1	x2	y1	y2
5%	665	652	2250	2100
3%	665	652	490	510
1%	665	652	3150	2900

3.2.3. GdVO₄ Luminescence Spectrometry of Yb³⁺ and Er³⁺ at Different Temperatures

- *GdVO₄ luminescence spectrometry of Yb³⁺ and Er³⁺ at 800 °C.*

With respect to **Figure 8** and **Table 7**, the highest luminescence peak belongs to the 1% crystal, having a wavelength of 523 nm and an absorbance of 2800 AU, followed by the 3% crystal with the second highest luminescence peak with a wavelength of 523 nm and an absorbance of 2600 AU. It is evident that the luminescence results for the 5% crystal turned out to be minimal compared to the 3% and 5% crystals.

- *GdVO₄ luminescence spectrometry of Yb³⁺ and Er³⁺ at 1000 °C.*

Looking at **Figure 9** and **Table 8**, we notice that the two highest peaks in erbium luminescence at 1000°C are for the 3% crystal, as it obtained absorbance results of 3050 AU with a wavelength of 523 nm, and the second one with an

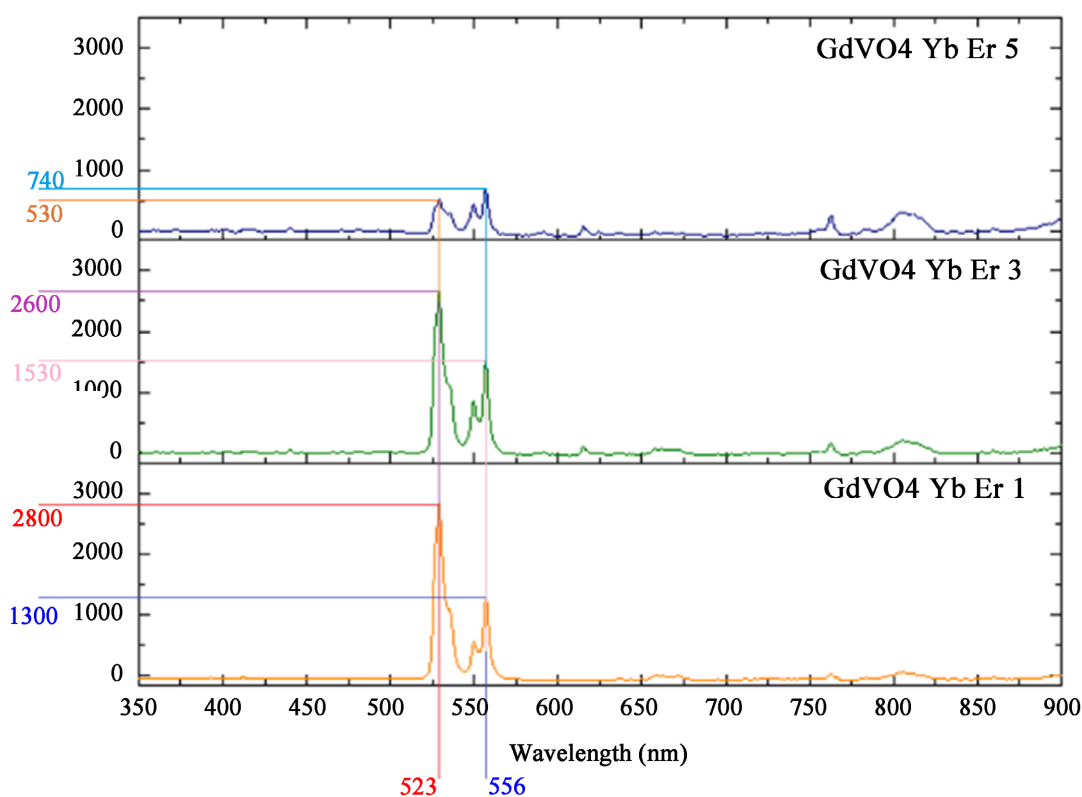


Figure 8. GdVO₄ luminescence spectrometry of Yb³⁺ and Er³⁺ at 800 °C.

Table 7. Wavelength and absorption of the highest GdVO₄ spots of Yb³⁺ and Er³⁺ at 800 °C.

Crystallite size	Wavelength (nm)		Absorption (U.A)	
	x1	x2	y1	y2
5%	523	556	530	740
3%	523	556	2600	1530
1%	523	556	2800	1300

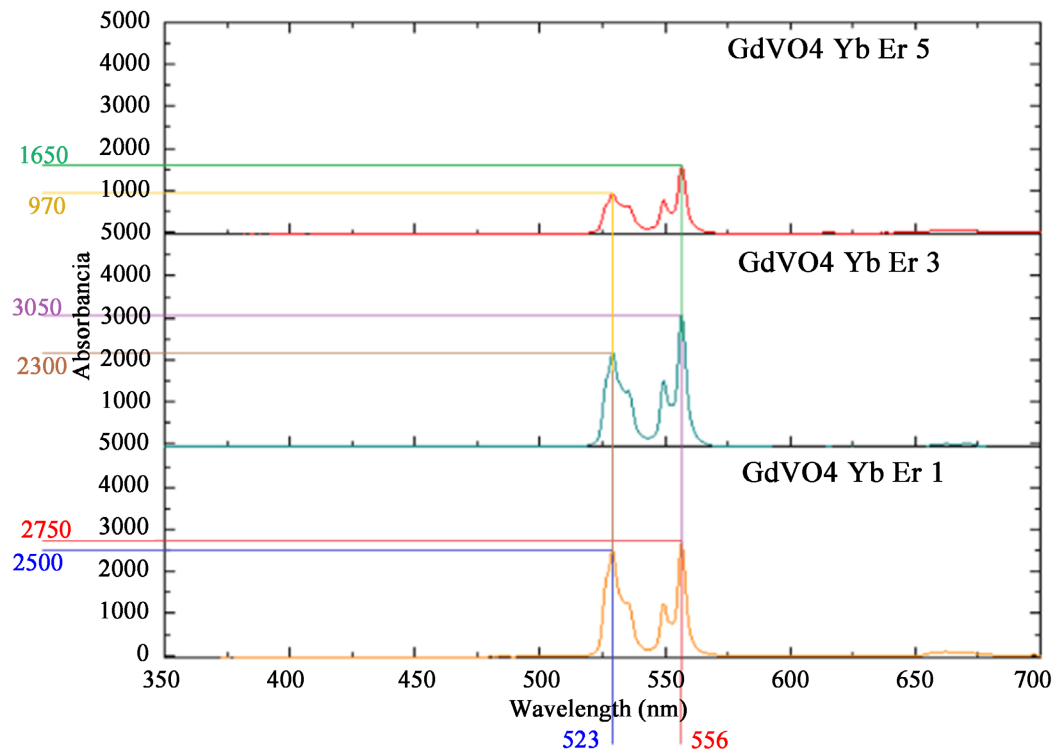


Figure 9. GdVO₄ luminescence spectrometry of Yb³⁺ and Er³⁺ at 1000°C.

Table 8. Wavelength and absorption of the highest GdVO₄ spots of Yb³⁺ and Er³⁺ at 1000°C.

Crystallite size	Wavelength (nm)		Absorption (U.A)	
	x1	x2	y1	y2
5%	523	556	970	1650
3%	523	556	2300	3050
1%	523	556	2500	2750

absorbance of 2300 AU and a wavelength of 556 nm.

Undoubtedly, the 5% crystal continues to present the smallest peaks compared to the 1% and 5% crystals.

It can be concluded that a GdVO₄ luminescence spectrometry of ytterbium (Yb³⁺) and Erbium (Er³⁺) has better results when the temperature is elevated, being the best option the 1% crystal at a temperature of 800°C, and for a temperature of 1000°C, the best option turned out to be the 3% crystal. While, in both cases, the 5% crystal presents the lowest luminescence.

3.3. Scanning Electron Microscopy

With the help of the JEOL microscope, scanning electron microscopy images were obtained to analyze the morphological evolution of the doped and undoped GdVO₄ powders that were heat treated at 900°C.

Figure 10 shows that the morphology of the GdVO₄ particles corresponds to

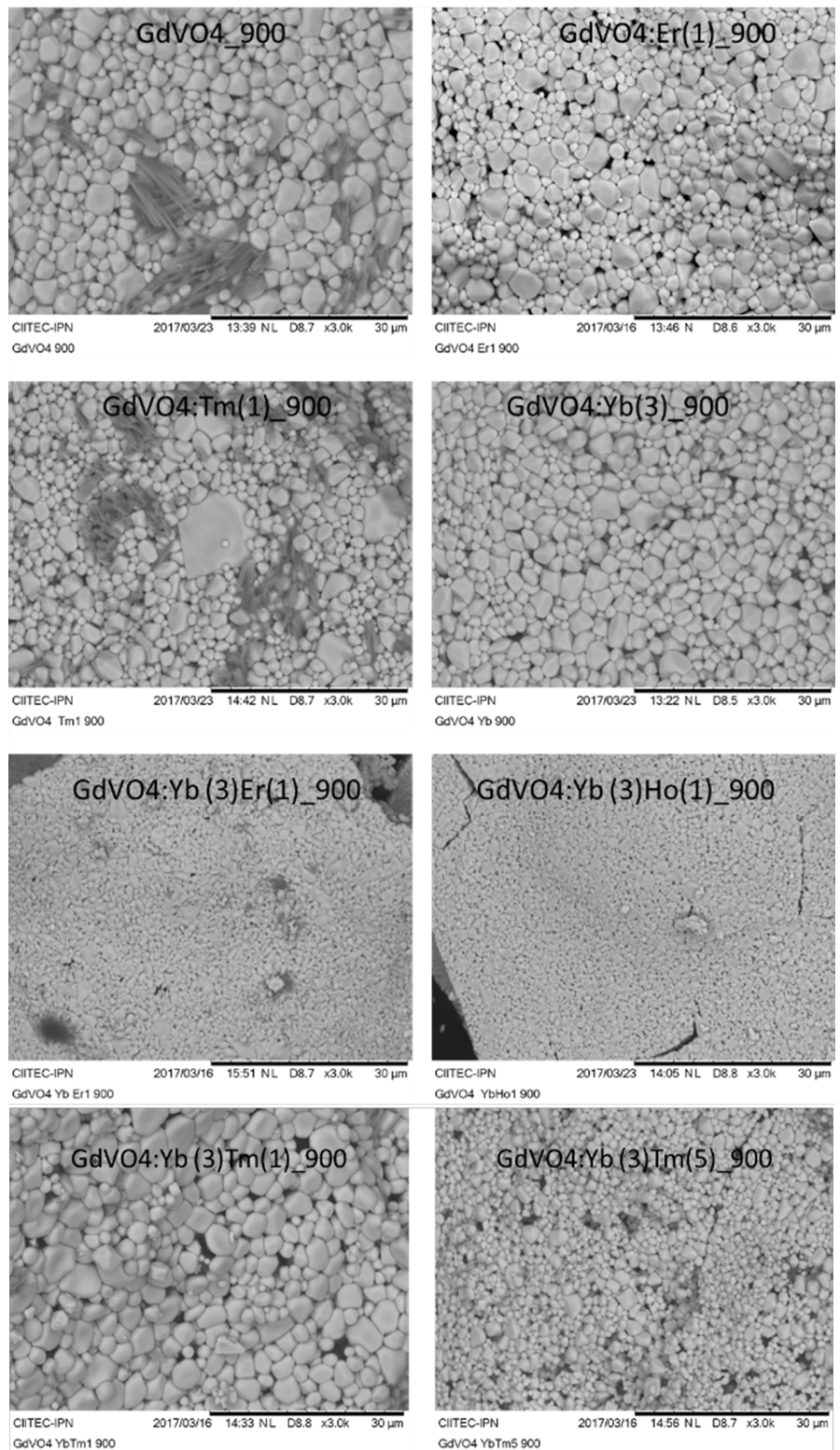


Figure 10. Morphology of GdVO₄ powders without rare earth, and with single and co-doped rare earth at 900°C heat treatment.

quasi-spherical particles, well densified and without any trace of porosity. In addition, it is shown that the particle size distribution is around 3 µm, although it

is to be noted also, the presence of fibers of approximately 12 μm in length. Due to the heat treatment conditions (900°C) of the particles begin to agglomerate and join together, causing a growth in particle size. Therefore, it is appreciated that there is not a closed distribution in the particle size, but rather, some of them are smaller than 3 μm in size.

Similarly, single-doped Scanning Electron Microscopy images were obtained with Erbium, Tullium and Iterbium and at 1% at., also presented in **Figure 10**. The images show a particle size distribution between 2 and 3 μm . From the above, it can be concluded that the concentration of dopant ions does not abruptly modify their morphology.

However, the presence of two dopants in the crystal structure of the GdVO_4 matrix causes the particle size to decrease drastically but not abruptly to values of about 1 μm . Therefore, the dopants are believed to retard grain growth.

4. Conclusions

The obtaining of luminescent results with doping of Yb^{3+} represents an advance in the research developed of this rare earth since in other studies the percentage of dopant is around 30% at, if it is possible to reduce the percentage of doping and to raise the energy efficiency in the up-conversion systems it would represent an advance in the way of the optimization of rare earths.

The images obtained by SEM show that the $\text{GdVO}_4:\text{Yb}^{3+}$, X^{3+} ($\text{X} = \text{Tm}, \text{Er}, \text{Ho}$) systems are spherical and semi-spherical, this is mainly due to the sol-gel process and the thermal treatment used, the spheres in the powders are spherical and semi-spherical. XRD and Spectroscopy results guarantee the formation of the proposed system, the synthesis method produced gadolinium vanadate, the FTIR showed the vibrations of the molecules and the spectroscopy result points out the influence of dopants on the system.

The results obtained confirmed a highly luminescent efficiency in up conversion, for the ions studied in this work.

Conflicts of Interest

The authors declare no conflicts of interest regarding the publication of this paper.

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