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Hydrothermal synthesis and luminescent properties of GdVO₄:Eu³⁺ nanophosphors

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Eu³⁺-doped GdVO₄ has been synthesised via hydrothermal method by altering the hydrothermal temperature, reaction time and surfactant. The microstructure and morphology information of the phosphors were investigated via the techniques of X-ray powder diffraction and scanning electronic microscopy, which show that the phosphors wear tetragonal phase and the products present various regular morphologies under different reaction conditions such as bulk and nanoparticle. Moreover, the morphologies of the products have been controlled by altering reaction temperature. In addition, the surfactant was also included to control the morphologies of the products and the phosphors present different morphologies. All the phosphors exhibit the characteristic fluorescence of Eu ion (⁵D₀ → ⁷F₂ and ⁵D₀ → ⁷F₁). The electric dipole transition ⁵D₀ → ⁷F₂ of Eu³⁺ is dominant indicating that most sites of Eu³⁺ ions in GdVO₄ have no inversion centre. Furthermore, we found that the reaction time and the morphologies have great influence on optical properties.

Keywords: hydrothermal synthesis; gadolinium vanadate; europium ion; photoluminescence

1. Introduction

Rare earth luminescent materials have considerable practical applications in almost any devices involving the artificial production of light, such as cathode ray tubes, lamps and X-ray detectors [1–7]. Europium ion is widely used as luminescent centre in much phosphors for the exhibited characteristic red emission corresponding to its ⁵D₀ → ⁷F₂ transition [8,9]. For example, during the past, the Eu³⁺-activated YVO₄ attracted attentions because of its broad application [10]. However, due to certain imperfect performance in luminescent intensity and colour purity of the current commercial phosphors much continuous work on the development of new high efficient phosphors still needs to be carried out. Recently, YVO₄ based phosphors have been synthesised by solid-state reaction methods [11–14], spray pyrolysis technique [15] and *in situ* co-precipitation process [16]. In addition to this, some researchers have prepared the nanoparticle of vanadate using co-precipitation and hydrothermal methods [17,18].

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As for the hosts, gadolinium vanadate (GdVO_4) is also a promising material for rare earth-activated oxide phosphors and near-infrared lasers. $\text{GdVO}_4:\text{Eu}^{3+}$ used as laser material has been extensively studied [19,20]. In the field of luminescent material, phosphors based on gadolinium compounds also play an important role because the Gd^{3+} ion has its lowest excited levels at relatively high energy, which is due to the stability of the half-filled shell ground state [21,22]. Traditional synthesis methods for rare earth vanadate luminescent materials were solid-state reactions at temperatures above 1300 K or simple wet-chemical technologies, such as chemical co-precipitation, sol-gel and colloidal reactions [23,24].

It is well known that the macroscopic properties of functional material systems with optical, magnetic and electronic properties are highly dependent on the sizes, morphologies, crystal types and compositions [25–28]. The materials may furthermore be of great application for their novel properties induced by reduced dimensionalities. Therefore, there are many reports on the synthesis of nano and microscaled phosphors of one-dimensional (1D) structures, such as wires or rods, tubes, coaxial cables and belts or ribbons, and of complex architectures based on 1D structure, such as branches, urchins and networks [29–32]. Thermal evaporation, laser ablation, metalorganic chemical vapour deposition (MOCVD) and molecular beam epitaxy (MBE) have been used to control the size and morphologies of materials [33–35]. The best solution to control the crystal size and morphology is to employ soft chemistry routes and in particular the hydrothermal processes, which are employed widely in the synthesis of rare earth ions – doped inorganic compounds. For instance, yttrium vanadate, lanthanum fluoride, lanthanum phosphate and yttrium oxide nanoparticles have been successfully prepared via this method [36–38].

In this article, we describe the synthesis and the luminescent properties of $\text{GdVO}_4:\text{Eu}^{3+}$ via the facile, mild, reproducible and easily controlled hydrothermal route. It is well known that the reaction temperature and time have great influence on the sizes and morphologies of the products. Herein, we have obtained different morphologies of luminescent materials by altering reaction temperature and time or adding surfactant. Moreover, it was found that all the factors influenced the final morphologies of the products. At the same time, we have compared the luminescent properties of these phosphors prepared under different conditions, indicating that the luminescent intensities are dependent on sizes and morphologies of the products.

2. Experimental section

2.1. Preparation of $\text{GdVO}_4:\text{Eu}^{3+}$

The initiative materials were Gd_2O_3 , Eu_2O_3 and NH_4VO_3 . First, Eu_2O_3 and Gd_2O_3 were added into concentrated nitric acid and heated until they were roapy to obtain $\text{Gd}(\text{NO}_3)_3$ and $\text{Eu}(\text{NO}_3)_3$. Then $\text{GdVO}_4:\text{Eu}^{3+}$ phosphors were synthesised under different conditions via the hydrothermal process, which is described as follows: $\text{Gd}(\text{NO}_3)_3 \cdot 6\text{H}_2\text{O}$ (2.0 mmol/0.902 g) and $\text{Eu}(\text{NO}_3)_3 \cdot 6\text{H}_2\text{O}$ (0.10 mmol/0.045 g) obtained above were mixed with appropriate amount of NH_4VO_3 (2.0 mmol/0.234 g) in distilled water to form the suspension liquid, and then the pH value was adjusted to 12. Subsequently, the solution was transferred into a Teflon lined stainless steel autoclave. Then the autoclave was sealed and maintained at 170°C for 3 days and 2 days, respectively and then cooled to room temperature in air. The resulting $\text{GdVO}_4:\text{Eu}^{3+}$ products were filtered, washed with

distilled water and absolute alcohol thrice to remove ions possibly remaining in the final products, and finally dried at 80°C in air for further characterisation.

At the same time, the same process was employed in the synthesis of $\text{GdVO}_4:\text{Eu}^{3+}$ products at 100°C for 3 days. Besides, by adding the surfactant the products wearing different morphologies have been prepared. The process is described as follows: $\text{Gd}(\text{NO}_3)_3 \cdot 6\text{H}_2\text{O}$ (2.0 mmol/0.902 g) and $\text{Eu}(\text{NO}_3)_3 \cdot 6\text{H}_2\text{O}$ (0.10 mmol/0.045 g) were mixed with appropriate amount of NH_4VO_3 (2.0 mmol/0.234 g) in distilled water, and then the surfactant was added, stirred for about an half hour to form the suspension liquid and other steps were similar to the above process.

2.2. Characterisation

The X-ray powder diffraction (XRD) patterns of all samples were performed on a Bruke/D8-Advance with CuK radiation ($\lambda = 1.518 \text{ \AA}$). The operating voltage and current were maintained at 40 kV and 40 mA, respectively. The scanning electronic microscopic images were obtained with Philips XL-30. The excitation and emission spectra were recorded with a Perkin-Elmer LS-55 model fluorometer equipped with a 150 W xenon lamp as the excitation source. Fourier transform infrared (FTIR) spectroscopy analysis was carried out using KBr discs in the region $4000\text{--}400 \text{ cm}^{-1}$ by using FTIR–Bruker–EQUINOX–55 under ambient condition. Ultraviolet-visible absorption spectra of the powder were recorded with a lambda 40 spectrometer (Perkin-Elmer). All the measurements were performed at room temperature.

3. Results and discussion

Figure 1 presents the selected XRD patterns of $\text{GdVO}_4:\text{Eu}^{3+}$ phosphor via the hydrothermal synthesis with (A) or without (B) CTAB surfactant. The X-ray-diffraction peaks of both the products are consistent with the literature data in the JCPDS file (No.17-0260), indicating that we obtained the $\text{GdVO}_4:\text{Eu}^{3+}$ powders with tetragonal phase, belonging to space group $141/amd(141)$ (lattice constants: $a = 7.213 \text{ \AA}$, $b = 7.213 \text{ \AA}$, $c = 6.367 \text{ \AA}$). The calculated size of the nanoparticles is 45–80 nm using Scherrer's equation, which delegates the dimension and is consistent with the scanning electron microscopy (SEM) observation. By comparing the sample A with CTAB and the sample B without CTAB, no apparent distinction could be found. The crystal phase did not change much under different reaction conditions, and all the curves exhibited very similar characters, so we just gave XRD patterns selectively. The same microstructure may lead to similar morphology.

We also examined the selected FTIR spectra of the $\text{GdVO}_4:\text{Eu}^{3+}$ phosphor (Figure 2). The bands at 3450 cm^{-1} and 1635 cm^{-1} are assigned to O–H stretching vibration and H–O–H bending vibration, respectively [39]. The two bands are the characteristic vibrations of water from air, physically absorbed on the sample surface, which is completely different from coordinated water in compounds. The V–O stretching vibration bands are designed at the $950\text{--}880 \text{ cm}^{-1}$ region and the O–V–O asymmetric vibration band at about 560 cm^{-1} .

The morphologies of the samples were obtained by SEM. Figure 3 shows the SEM images of $\text{GdVO}_4:\text{Eu}^{3+}$ phosphors ((a) and (b) under heating at 170°C for 2 and 3 days, respectively). It can be seen that all the products mainly consist of solid microcrystalline

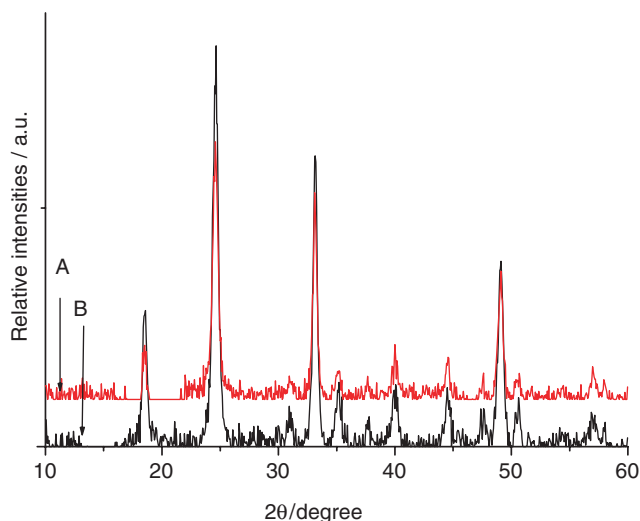


Figure 1. Selected XRD pattern of $\text{GdVO}_4:\text{Eu}^{3+}$ phosphor at 170°C for 3 days: (A) with 0.06 g CTAB and (B) without CTAB.

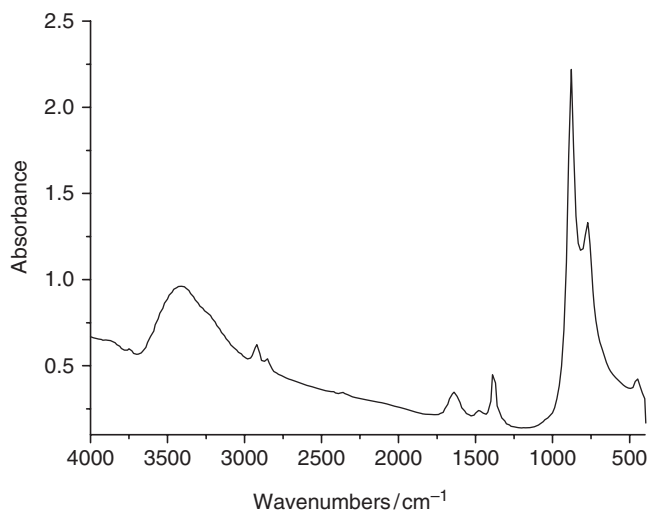


Figure 2. Selected IR spectrum of $\text{GdVO}_4:\text{Eu}^{3+}$ phosphor at 170°C for 3 days with 0.1 g CTAB.

structures, which show some conglomeration phenomena. While the reaction temperature was 170°C , the grain size is not homogeneously dispersed. At the same time, we also synthesised $\text{GdVO}_4:\text{Eu}^{3+}$ phosphors under 100°C , the SEM of which is shown in Figure 3(c). From Figure 3(b) and (c), it can be seen that the products present bulk morphology conglomerating with nanoflake and the particle size did not change much with altering temperature. It is well known that any change in the hydrothermal conditions, such as solvents, time, pH value, reaction temperature, will change the size and

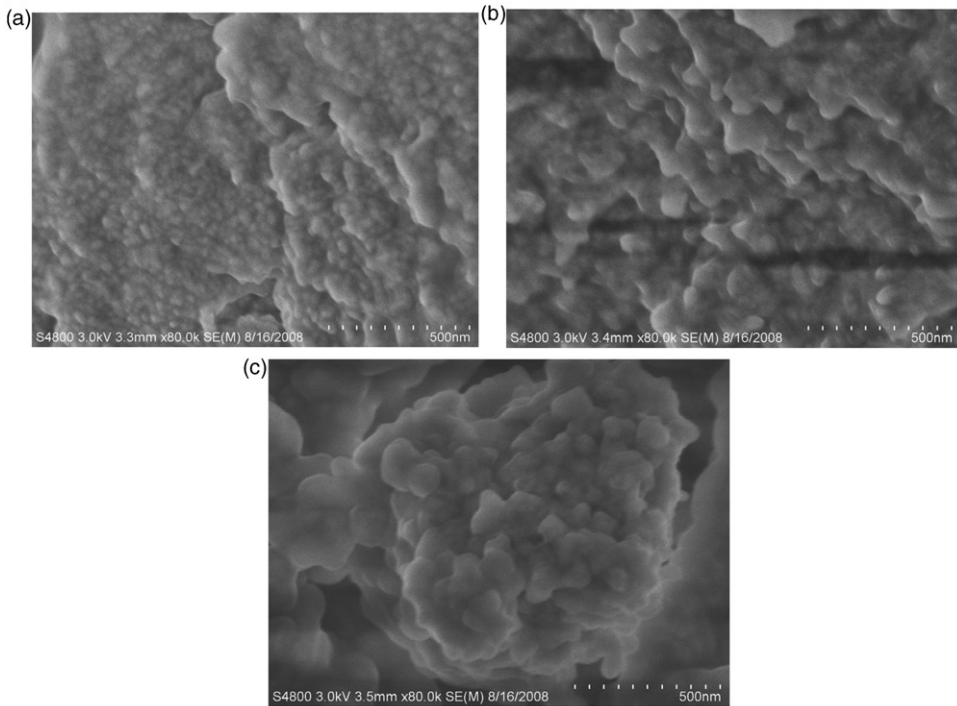


Figure 3. Selected SEM images of $\text{GdVO}_4:\text{Eu}^{3+}$ phosphors: (a) 170°C , 2 days; (b) 170°C , 3 days and (c) 100°C , 3 days.

morphology of the product. From the above discussion, we can conclude that the influence of both reaction temperature and reacting time is not obvious. The picture shows some nanoparticles with particle size 50–100 nm, which is in agreement with the estimation from XRD.

During the hydrothermal process, the control in the particle size and morphologies not only depend on the inherent structural characteristics of the compounds but also on external experimental conditions, including reaction time, reaction temperature, surfactants and solvents. Therefore, the surfactant is also a key factor for controlling the morphologies of the products (Figure 4(a), (b) and (c)). The product presents the different morphologies heated at 170°C for 3 days in the presence of CTAB. In the hydrothermal process, the surfactants can be used as templates; they may form the spherical micelles and then play the role of the template leading to the products with the nanoparticle morphologies. As a result, the product obtained in the presence of the surfactant shows nanograin morphologies. The morphology of a crystal is the result of the relative growth rates of its different faces, the general rule being that the faces, which grow very slowly, are expressed in the crystal habit. The growth rates of the various crystal faces are determined by intermolecular interactions between molecules in the crystal as well as by a number of external parameters such as solvent, degree of supersaturating, duration, temperature and foreign materials. Any one of these may lead to dramatic modifications in crystal morphology [40].

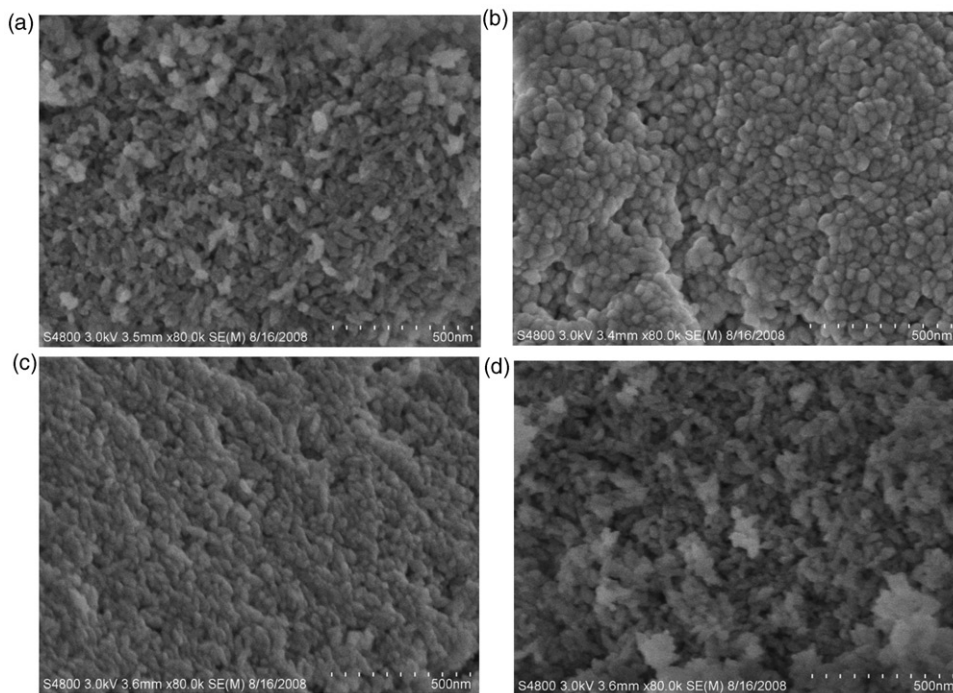


Figure 4. Selected SEM images of $\text{GdVO}_4:\text{Eu}^{3+}$ phosphors: (a) 170°C , 0.2 g CTAB, 3 days; (b) 170°C , 0.06 g CTAB, 3 days; (c) 170°C , 0.1 g CTAB, 2 days and (d) 100°C , 0.1 g CTAB, 2 days.

The absorption spectra of $\text{GdVO}_4:\text{Eu}^{3+}$ phosphors have been examined (Figure 5(a)), and involve several bands. The strong band peaking at 280 nm was ascribed to the absorption band of charge transfer band (CTB) (O–Eu) and a weaker band originating from the Eu^{3+} f–f transitions was also observed in the region 300–400 nm, which agreed with the excitation spectra. Besides, in the visible-light region negative absorption of 610 nm is due to the $^5\text{D}_0 \rightarrow ^7\text{F}_2$ transition of Eu^{3+} ion. The different morphologies of the phosphors may have influence on the optical properties. The excitation mission and emission spectra of $\text{GdVO}_4:\text{Eu}^{3+}$ phosphors are shown in Figure 5(b). All the excitation spectra of $\text{GdVO}_4:\text{Eu}^{3+}$ phosphors were taken at an emission wavelength of 613 nm. All the curves exhibited very similar characters, so we just gave one excitation spectrum selectively (Figure 5(b)). A broad band was observed in the shorter wavelength region from 220 to 280 nm in the excitation spectrum, originating from a charge transfer (CT) transition, as shown in Figure 5(b). This broad band took place by electron delocalisation from an oxygen 2p orbital to an empty 4f orbital of europium ion. It was reported that the peak position of CTB was highly dependent on the Eu–O bond length [41,42]. Besides, the sharp lines in the longer wavelength of 300–400 nm were observed in the spectrum, corresponding to direct excitation of the Eu^{3+} ground state to higher levels of the 4f-manifold, and their assignments are marked in the figure according to the previous work [43].

The emission spectra of all Eu^{3+} activated phosphors have been involved in the following emission lines: $^5\text{D}_0 \rightarrow ^7\text{F}_1$, $^5\text{D}_0 \rightarrow ^7\text{F}_2$, $^5\text{D}_0 \rightarrow ^7\text{F}_3$ and $^5\text{D}_0 \rightarrow ^7\text{F}_4$, which are

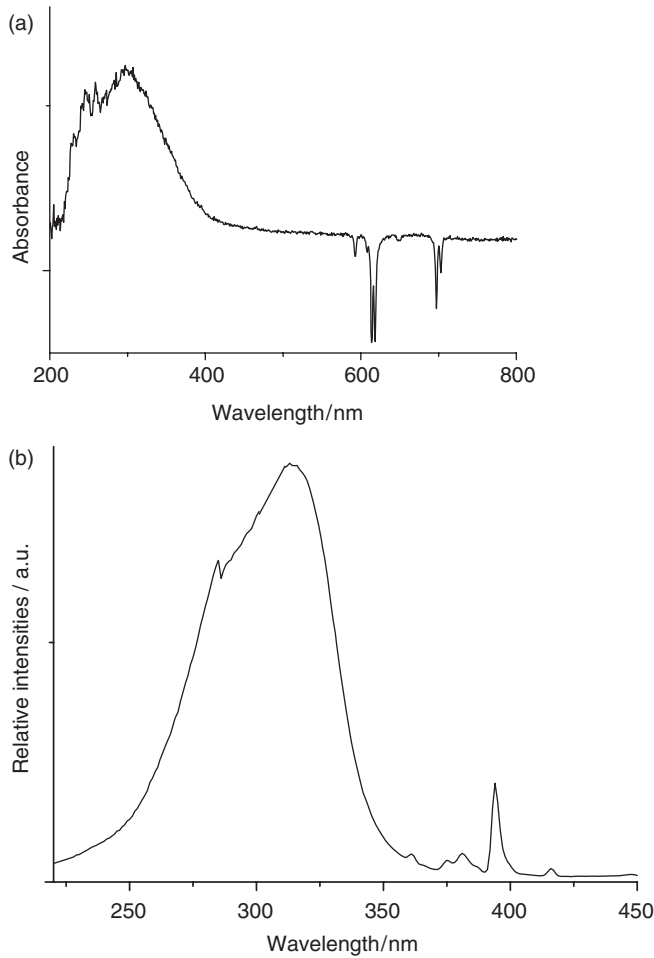


Figure 5. Selected diffuse reflectance ultraviolet visible spectrum (a) and excitation spectrum (b) of $\text{GdVO}_4:\text{Eu}^{3+}$ phosphor at 170°C for 3 days with 0.1 g CTAB.

determined by transitions between their f-electron energy levels (Figure 6). The orange–red emission lines at around 590 nm originate from the magnetic dipole transition ${}^5\text{D}_0 \rightarrow {}^7\text{F}_1$, while the ${}^5\text{D}_0 \rightarrow {}^7\text{F}_2$ lines originate from the electric dipole transition. From the emission spectra of $\text{GdVO}_4:\text{Eu}^{3+}$ phosphors, we can find that peak position of the emission lines are almost the same, but the intensity patterns of their luminescence spectra show some small differences. According to the Judd–Ofelt theory, the magnetic dipole transition is permitted. However, the electric dipole transition is allowed only when the europium ion occupies a site without an inversion centre and the intensity is significantly affected by the symmetry in local environments around Eu^{3+} ions [44]. If the Eu^{3+} ions occupy an inversion symmetry site in the GdVO_4 crystal lattice, the orange–red emission, magnetic transition ${}^5\text{D}_0 \rightarrow {}^7\text{F}_1$ (around 590 nm), is the dominant transition. In contrast, the electric

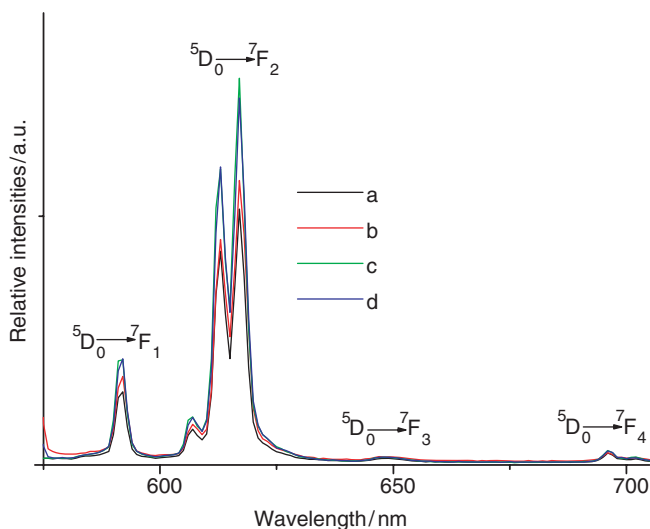


Figure 6. Emission spectra of $\text{GdVO}_4:\text{Eu}^{3+}$ phosphors at 170°C for 3 days with different CTAB: (a) 0.2 g CTAB; (b) 0.4 g CTAB (c) 0.6 g CTAB and (d) 0.1 g CTAB.

dipole transition ${}^5\text{D}_0 \rightarrow {}^7\text{F}_2$ (around 610–620 nm) is the dominant transition. According to the emission spectra, it can be concluded that most Eu^{3+} ions have no inversion centres and the electric dipole transition ${}^5\text{D}_0 \rightarrow {}^7\text{F}_2$ is dominant. Due to the interaction between Eu^{3+} and O^{2-} ions in GdVO_4 doped with Eu^{3+} , level split can be caused by the crystal field and a little split of Eu^{3+} emission bands has been observed. From Figure 7(a), it can be seen that the optical property is enhanced when the amount of the surfactant increases. We surmised that the result is relative to the morphologies and sizes of the products. The three-dimensional sizes of crystalline grain are very thick to afford high strength. It needs to be referred that the crystalline powder and micrometre dimension for these powders with high strength would be very useful for the application to obtain high efficient phosphors because these microcrystalline materials can result in high luminescent intensities [45]. We also investigated the influence of the reaction time on the optical property (Figure 7(b)) and found that the reaction time has no obvious influence on the optical property. This can be ascribed to the similar morphologies of the phosphors.

4. Conclusions

In summary, Eu^{3+} -doped gadolinium vanadium oxides ($\text{GdVO}_4:\text{Eu}^{3+}$) with tetragonal phase have been synthesised via hydrothermal method. The products present various regular morphologies under different reaction conditions such as bulk, nanorod and nanoparticle. Moreover, the morphologies of the products have been controlled successfully by altering reaction temperature. The surfactant was also included to control the morphologies of the products and the presence of surfactant can avoid conglomeration phenomena. All the phosphors exhibit the characteristic fluorescence of Eu ion. The electric dipole transition ${}^5\text{D}_0 \rightarrow {}^7\text{F}_2$ of Eu^{3+} ions is dominant indicating that most sites of

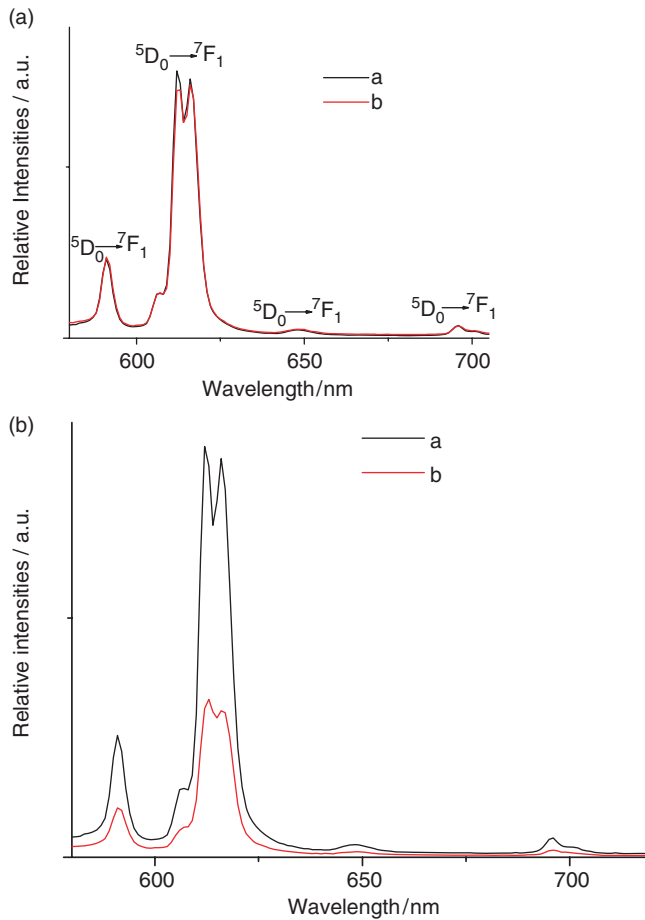


Figure 7. Selected emission spectra of GdVO₄:Eu³⁺ phosphors: (a) 170°C for different time. (a) 2 days; (b) 3 days and (b) at different temperature for 3 days (a) 170°C; (b) 100°C.

Eu³⁺ ions in GdVO₄ have no inversion centre. Furthermore, we found that the relatively small particle size and regular morphology are favourable for the optical properties.

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