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Color-tunable nanophosphors by co-doping flame-made Y_2O_3 with Tb and Eu

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Abstract

Rare-earth phosphors with tunable optical properties are used in display panels and fluorescent lamps and have potential applications in lasers and bio-imaging. Here, non-aggregated Y_2O_3 nanocrystals either doped with Tb^{3+} (1-5 at%) or co-doped with Tb^{3+} (2 at%) and Eu^{3+} (0.1-2 at %) ions are made in one-step by scalable flame spray pyrolysis. The morphology of these nanophosphors is investigated by X-ray diffraction, electron microscopy and N_2 adsorption while their optical properties are monitored by photoluminescent spectroscopy. When yttria nanocrystals are doped with terbium, a bright green emission is obtained at an optimum Tb-content of 2 at%. When, however, europium is added, the emission color of these Tb-doped yttria nanophosphors can be tuned precisely from green to red depending on the Tb/Eu ratio. Furthermore, energy-transfer from Tb^{3+} to Eu^{3+} is observed, thus allowing the control of the excitation spectra of the co-doped nanophosphors.

Keywords

nanoparticles; light emitting materials; yttrium oxide; energy transfer

1. Introduction

Rare-earth phosphors are light emitting materials that find a wide variety of applications.^[1] These materials are multicomponent: one material being the host matrix and the rare-earth doping element being responsible for the radiation.^[2] The emission wavelength of such materials depends mostly on the chosen doping rare-earth element. Phosphors are commonly used in fluorescent lamps and luminescent displays^[1] and have potential applications in lasers^[3] and X-ray imaging.^[4] Nanosized phosphors (nanophosphors) improve the resolution of displays^[5] and have promising applications as bio-imaging probes.^[6] The superior photostability (no blinking or fading) and biocompatibility^[7] of nanophosphors makes them advantageous over traditionally used organic dyes and semiconducting nanoparticles. Organic fluorescent dyes photobleach during bio-imaging while semiconducting nanoparticles exhibit optical blinking^[8] and can be toxic.^[9] Furthermore, the nano size of dispersible nanophosphors facilitates their employment in nanocomposite materials such as flexible displays.^[5]

One of the most studied ceramics as host matrix for phosphors is yttrium oxide (Y_2O_3). Several studies address synthesis of nanosized, Y_2O_3 -based phosphors doped with Eu^{3+} ions for their bright red emission. Such phosphor nanoparticles are typically made by elaborate

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wet methods^[7,10,11] while it is not unusual that further annealing process steps are required to obtain the desired crystallinity.^[12,13] Post heat-treatment of such nanoparticles, however, results in aggregates with strong sinter necks that may hinder their dispersion in host polymers or liquid suspensions.^[14] An alternative to wet-chemistry methods is flame aerosol synthesis^[15-18] which is a scalable process^[19] that allows for fine tuning of the crystallinity of the resulting particles in one-step without any post heat-treatment.^[17] This results in mostly non-aggregated nanoparticles that are attractive in bio-imaging.^[6]

The color tunability of Y_2O_3 -based nanophosphors can be achieved by doping with rare-earth elements.^[1] For example, while Eu^{3+} doping of Y_2O_3 results in a red emission, doping it with Tb^{3+} results in a green emission.^[10] In fact, this color tunability is exploited already by combining three different colored phosphors (blue, green, red) for white-light lamps.^[1] For all rare-earth phosphors, there is an optimum concentration of the doping material.^[20] This concentration, however, depends on several parameters, such as synthetic route or even the size of the phosphors.^[12]

Even though there are many studies investigating synthesis and optical properties of Y_2O_3 -based nanophosphors doped with a rare-earth element, there are only a few that explore the co-doping of two or more rare-earth elements in the same crystal host matrix. Co-doping enables the fine tuning of the excitation and emission spectra of phosphors.^[21] When Y_2O_3 is co-doped with Eu and Tb, for example, a strong energy transfer occurs from Tb^{3+} to Eu^{3+} ions,^[21] while a back energy-transfer from Eu^{3+} to Tb^{3+} is not significant.^[13] In fact, there is an optimum ratio ($\text{Eu}/\text{Tb} = 8$) for maximum luminescent efficiency.^[22] Such energy transfer has been observed also for other rare-earth doped up-converting nanophosphors (e.g. Yb-Er).^[7]

Here, the one-step synthesis of $\text{Y}_2\text{O}_3:\text{Tb}^{3+}$ and co-doped $\text{Y}_2\text{O}_3:\text{Tb}^{3+}/\text{Eu}^{3+}$ nanoparticles is explored by flame aerosol technology.^[17] The morphology and particle characteristics are investigated by EDX spectroscopy, TEM analysis, X-ray diffraction and N_2 adsorption (BET). The optical properties of the as-prepared nanophosphors are explored by photoluminescent spectroscopy. The optimum Tb-content is investigated by monitoring the photoluminescent intensity of the $\text{Y}_2\text{O}_3:\text{Tb}^{3+}$ nanophosphors. The emission wavelength of the co-doped nanophosphors is tuned by the ratio of the doping rare-earth elements. A strong energy transfer is observed from Tb^{3+} to Eu^{3+} which allows for the characteristic red emission of Eu^{3+} at higher excitation wavelengths.

2. Experimental section

Multi-component nanophosphors were produced by flame spray pyrolysis (FSP) of appropriate precursor solutions as described in detail elsewhere.^[17] Yttrium nitrate (Aldrich, 99.9%) was dissolved in a 1:1 by volume mixture of 2-ethylhexanoic acid (EHA, Riedel-de Haen, 99%) and ethanol (Alcosuisse) to form the precursor solution. The molarity was kept constant at 0.5M for Y metal. The terbium and/or europium doping was achieved by adding 1-5 at% terbium nitrate (Aldrich, 99.9%) or 0.1-2 at% europium nitrate (Fluka, 95%) to the above solution. The atomic fraction (at%) of dopants was defined with respect to the total metal ion concentration. The precursor solution was fed to the FSP nozzle at a constant feed rate (11.6 mL/min) provided by a syringe pump (Inotech) and dispersed to a fine spray by 3 L/min oxygen (PanGas, purity >99.9%). The pressure drop at the nozzle tip was kept constant at 1.5 bar. Subsequently, the spray was ignited by a premixed methane/oxygen (1.5/3.2 L/min, PanGas, purity >99.9%) flame leading to formation of nanophosphor particles that were collected on a glass microfiber filter (Whatman GF6, 257 mm diameter) with the aid of a vacuum pump (Busch, Seco SV 1040C).

X-ray diffraction (XRD) patterns were recorded by a Bruker AXS D8 Advance diffractometer (40 kV, 40 mA, Cu K α radiation) from $2\theta=20-70^\circ$ with a step size of 0.03° . The obtained spectra were fitted using the TOPAS 3 software (Bruker) and the Rietveld fundamental parameter refinement.^[17] The specific surface area (SSA) was obtained according to Brunauer-Emmet-Teller (BET) by five-point N $_2$ adsorption at 77 K (Micrometrics Tristar 3000). Prior to that, samples were degassed in N $_2$ for at least 1 h at 150°C. High resolution transmission electron microscopy (HRTEM) was performed with a CM30ST microscope (FEI; LaB6 cathode, operated at 300 kV, point resolution ~ 2 Å). The electron beam could be set to selected areas to determine material composition by energy dispersive X-ray spectroscopy (EDXS). Product particles were dispersed in ethanol and deposited onto a perforated carbon foil supported on a copper grid. The photoluminescence of the produced particles was characterized at room temperature using a fluorescence spectrophotometer (Varian Cary Eclipse) containing a Xe flash lamp with tunable emission wavelength. Samples of 30 mg were filled in a cylindrical substrate holder of 10 mm diameter and pressed towards a quartz glass front window. Emission spectra were recorded from 450 – 650 nm, excitation spectra from 200 – 400 nm with a step size of 0.5 nm. Additionally, photoluminescence decay curves were recorded at a resolution of 0.03 ms. The obtained data were fitted by the Cary Eclipse software (Varian) and thus first order exponential decay time constants were extracted.

3. Results and Discussion

Phosphor Morphology

Energy-dispersive X-ray (EDX) spectroscopy over a large area of the 2 at% Tb-doped Y $_2$ O $_3$ sample (Figure 1a) reveals the presence of both Y and Tb. The C and Cu peaks come from the carbon-coated copper grid which was used to obtain the TEM images. Figure 1b shows a TEM image of the same nanoparticles that are non-aggregated.^[17] A high resolution TEM image showing a single crystalline nanoparticle is shown in Figure 1c. The distance between their crystal planes is 3.03 Å corresponding to the (222) crystal plane^[13] of cubic Y $_2$ O $_3$.

Figure 2 shows the X-ray diffraction spectra of as-prepared pure Y $_2$ O $_3$ nanoparticles that correspond to mostly cubic^[5] phase of Y $_2$ O $_3$, without involving post heat-treatment.^[17] The long residence time in high temperatures allows for control of the crystalline structure from the monoclinic phase for low combustion enthalpy flames to the cubic phase for high combustion enthalpy flames, as here.^[17] All XRD spectra in the presence of terbium doping (1-5 at%) are identical (not shown). The cubic mass fraction as estimated by Rietveld analysis is $\sim 90\%$ while the rest $\sim 10\%$ corresponds to the monoclinic phase. The presence of terbium doping does not influence significantly this crystallinity, as seen in the inset of Figure 2. Perhaps there is a slight decrease with an increasing Tb-content. This is consistent with flame-made Y $_2$ O $_3$ doped with other rare earth ions (e.g. Eu)^[5,17] where a slight decrease of the Y $_2$ O $_3$ cubic mass fraction was also observed for an increasing Eu-doping concentration.

The average crystal sizes of cubic ($d_{XRD,c}$, circles) and monoclinic ($d_{XRD,m}$, squares) Y $_2$ O $_3$ as determined by XRD are presented in Figure 3. The average particle or grain diameter as determined from N $_2$ adsorption (d_{BET} , triangles) is also shown in Figure 3. The monoclinic Y $_2$ O $_3$ particles are smaller than the cubic ones consistent with the literature.^[17] The slightly lower d_{BET} values than $d_{XRD,c}$ originate from the small fraction of the monoclinic Y $_2$ O $_3$ (~ 10 wt%). The presence of Tb does not influence significantly the morphology of the nanophosphors, consistent with prior flame-made Y $_2$ O $_3$ -based nanophosphors^[5,15-17] as well as with rare-earth doped Y $_2$ O $_3$ -based nanophosphors made by wet chemistry.^[7,12,13] The good agreement between $d_{XRD,c}$ and d_{BET} indicates that the particles are monocrystalline and non-aggregated, a desirable feature for bioimaging applications.^[7]

Photoluminescence of $\text{Y}_2\text{O}_3:\text{Tb}^{3+}$ nanophosphors

Figure 4a shows the excitation spectrum of the 2 at% Tb-doped Y_2O_3 monitored at 545 nm. It consists of two broad bands at ~280 and 310 nm corresponding to $4f \rightarrow 5d$ transition^[13] of Tb^{3+} . The emission spectrum of the same sample (2 at% Tb) under 276 nm excitation is shown in Figure 4b. There are the characteristic emission peaks attributed to Tb^{3+} ions^[7] with the most intense one at 545 nm (green) corresponding to the $^5\text{D}_4 \rightarrow ^7\text{F}_5$ transition.^[7,13]

The optimum Tb doping concentration was investigated by varying the Tb content from 1 to 5 at%. Figure 5 shows the phosphorescence maximum intensity of the $\text{Y}_2\text{O}_3:\text{Tb}^{3+}$ nanophosphors (at 545 nm) under 276 nm excitation as a function of the Tb-content (circles). The error bars correspond to the standard deviation of three different samples. The highest intensity is obtained for 2 at% Tb. At higher Tb contents, the intensity decreases. The enhanced photoluminescence (PL) for higher doping up to a critical concentration (here 2 at%) results from the increasing number of luminescent centers. The PL reduction for higher doping, the so-called “quenching”, is a characteristic behavior and inherent to all phosphors.^[20] It is based on energy transfer between adjacent luminescent centers.^[23] As the energy levels of similar lanthanide ions match perfectly, this energy transfer is highly efficient and will be favored instead of the light emitting decay.^[13] At high doping concentrations, this probability is enhanced.^[12] Even though $\text{Y}_2\text{O}_3:\text{Tb}^{3+}$ nanoparticles have never been made by flame spray pyrolysis before, the present optimum dopant concentration is within the reported ones from different synthetic routes.^[12,13]

The PL-decay time constant is shown in Figure 5 (triangles) also. For the nanophosphor with the highest emission intensity (2 at% Tb) it is 2.1 ms. For an increasing doping concentration, however, it monotonically decreases, reaching 1.25 ms for the 5 at% Tb, values similar to $\text{Y}_2\text{O}_3:\text{Tb}^{3+}$ nanophosphors made by wet chemistry.^[13,24] Such a reduction of the decay time for an increasing rare-earth ion concentration has been observed for $\text{Y}_2\text{O}_3:\text{Tb}^{3+}$ at similar Tb-contents (0.005-5 at%)^[25] as well as for flame-made $\text{Y}_2\text{O}_3:\text{Eu}^{3+}$ nanophosphors.^[5,26]

Co-doped $\text{Y}_2\text{O}_3:\text{Tb}/\text{Eu}$

Co-doping the nanophosphors with Eu and Tb did not alter the morphology and crystallinity of flame-made Y_2O_3 based nanophosphors (Figure 1). Figure 6 shows the emission spectra of the co-doped $\text{Y}_2\text{O}_3:\text{Tb}/\text{Eu}$ under 276 nm excitation (the wavelength with maximum intensity attributed to Tb^{3+} transitions, Figure 4). The Tb-content was kept constant at 2 at% while the Eu content varied from 0 to 2 at%. For comparison, also a sample in the absence of Tb was made (with 1 at% Eu)^[26]. Even with a minimal Eu content of 0.1 at%, the characteristic emission peak at 612 nm attributed to the $^5\text{D}_0 \rightarrow ^7\text{F}_2$ transition within Eu^{3+} ions^[7] is detectable. As the Eu-content increases, the corresponding intensity peak (at 612 nm) increases, as more Eu^{3+} luminescent centers are created. For the nanophosphor with 2 at% Tb and 0.5 at% Eu the emission peaks at 545 and 612 nm have similar intensities (for excitation at 276 nm). This is consistent with such co-doped nanophosphors made by wet-chemistry^[13] employing, however, higher dopant concentrations (6 at% Tb and 4 at% Eu).

Figure 7 shows suspensions of the as-prepared nanophosphors in ethanol under 254 nm excitation (from a commercial UV lamp). The change of the ratio between the two doping ions results in different colors. Therefore, by adjusting the ratio between the doping rare earth ions in the Y_2O_3 host matrix, a fine tuning of the emitted color can be achieved: from light green-blue for 2 at% Tb and yellow for 2 at% Tb/ 0.25 at% Eu, to red for 2 at% Tb/ 1 at% Eu.

A close examination of Figure 6 shows that the intensity of the Tb^{3+} emission peaks decreases for an increasing Eu-content, although the Tb-content remains constant. This is

seen also in Figure 8a where the excitation spectra monitoring the 545 nm emission peak at an increasing Eu-content are presented. This indicates that there is no radiative^[27] energy transfer^[13] from Eu^{3+} to Tb^{3+} . In contrast, the intensity of the Eu^{3+} peak at 612 nm for 1 at % Eu content is higher for the sample co-doped with Tb (Figure 6). The excitation wavelength of 276 nm with which the emission spectra of Figure 6 are recorded corresponds to the $4f \rightarrow 5d$ transition of Tb^{3+} (Figure 4). This indicates that there is a strong energy transfer^[13,22] from Tb^{3+} to Eu^{3+} . This energy transfer is further verified from the excitation spectra of the co-doped nanophosphors monitoring the 612 nm Eu peak (Figure 8b). In the absence of Tb (top line), the excitation band centered around 230 nm is typical for $\text{Y}_2\text{O}_3:\text{Eu}^{3+}$ nanophosphors.^[7] In the presence of Tb, however, there is Eu^{3+} emission at 612 nm after exciting at wavelengths corresponding to the Tb^{3+} ions transitions (bands at 280 and 310 nm). It should be noted, however, that the emitted color of each co-doped nanophosphor (such as the ones observed in Figure 7) depends on the excitation wavelength. Figure 8 also shows that when the excitation wavelength is <250 nm, only red emission at 612 nm occurs (e.g. 2 Tb/0.5 Eu), with no green emission at 545 nm. Therefore, it is possible to control the excitation spectrum, as well as the emission color of the co-doped $\text{Y}_2\text{O}_3:\text{Tb}^{3+}/\text{Eu}^{3+}$ nanophosphors through the energy transfer from Tb^{3+} to Eu^{3+} .

4. Conclusions

Multicomponent nanophosphors have been made in one-step by flame spray pyrolysis. Rare earth Tb^{3+} and Eu^{3+} ions have been incorporated in nanocrystalline Y_2O_3 (cubic phase). The resulting nanocrystals ($d_{XRD} = 33$ nm) were non-aggregated and no further post heat-treatment was needed. Photoluminescent examination revealed bright green phosphorescence for $\text{Y}_2\text{O}_3:\text{Tb}^{3+}$ nanocrystals in agreement with the literature. When these nanophosphors were co-doped with Eu^{3+} , their emission color could be controlled from bright green to bright red. A strong energy transfer occurs from Tb^{3+} to Eu^{3+} ions, which results in the precise tuning of the excitation spectrum of these light emitting nanophosphors. This fine tuning along with the scalable process employed here for synthesis of these nanophosphors may facilitate their employment in several applications, such as fluorescent lamps, display panels and bio-imaging given their photostability and biocompatibility.

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Figure 1.

Energy dispersive X-ray (EDX) spectrum of the 2 at% Tb-doped Y_2O_3 with its corresponding Tb and Y peaks. The C and Cu peaks come from the TEM carbon coated copper grid TEM. (b) A TEM image showing the non-aggregated structure of the 2 at% Tb-doped Y_2O_3 nanoparticles. (c) A high resolution TEM image showing the crystal planes of a single nanoparticle. The distance between crystal planes (3.03 Å) corresponds to the (222) crystal plane of Y_2O_3 .

Figure 2.

X-ray diffraction patterns of flame-made pure Y_2O_3 nanoparticles. All peaks correspond to cubic Y_2O_3 . As inset, the cubic Y_2O_3 mass fraction as a function of the Tb-content is presented. The presence of Tb does not significantly influence the crystallinity of the particles.

Figure 3.

The average cubic ($d_{XRD,cubic}$, circles) and monoclinic ($d_{XRD,monoclinic}$, squares) Y_2O_3 crystal sizes and the average particle or grain diameter as determined by N_2 adsorption (d_{BET} , triangles) of the as-prepared flame-made nanophosphors as a function of the Tb-content that has hardly any influence on them.

Figure 4.

(a) The excitation spectrum of the 2 at% Tb-doped Y_2O_3 nanoparticles monitored at 545 nm. The two bands around 280 and 310 nm are attributed to Tb^{3+} transitions. (b) Emission spectrum of the same sample under 276 nm excitation. The appearing peaks correspond to Tb^{3+} ion transitions, with most dominant being the one at 545 nm attributed to the $^5\text{D}_4 \rightarrow ^7\text{F}_5$ transition.

Figure 5.

The maximum phosphorescence intensity monitored at 545 nm under excitation of 276 nm (circles, left axis) and the decay time constant (triangles, right axis) as a function of Tb-content. The optimum Tb-concentration is 2 at%.

Figure 6. Emission spectra of the co-doped $\text{Y}_2\text{O}_3:\text{Tb}^{3+}/\text{Eu}^{3+}$ nanophosphors under 276 nm excitation. For an increasing Eu-content, the phosphorescence peaks attributed to Eu^{3+} (dominating one at 612 nm) are also increasing.

Figure 7.

Images of the co-doped $\text{Y}_2\text{O}_3:\text{Tb}^{3+}/\text{Eu}^{3+}$ nanophosphors dispersed in ethanol under 254 nm excitation. The color of the nanophosphors can be fine tuned by selecting the atomic ratio of these rare earth ions.

Figure 8.

Excitation spectra of the co-doped $\text{Y}_2\text{O}_3:\text{Tb}^{3+}/\text{Eu}^{3+}$ nanophosphors monitored at 545 nm (a) and 612 nm (b). The existence of the broad bands at 280 and 310 nm when monitoring the Eu^{3+} emission peak (612 nm) indicates an energy transfer from Tb^{3+} to Eu^{3+} ions.