Synthesis and photophysical properties of a highly luminescent Eu^{III}-containing hybrid thin film

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Abstract:

A luminescent thin film, Eu@glass, has been prepared on the surface of glass substrates from a silylated diamidopyridyl ligand **L** and the complex [Eu(dbm)$_3$(H$_2$O)$_2$] (Hdbm = dibenzoylmethane) via a sol-gel method. The Eu@glass exhibits intense red photoluminescence under the irradiation of near UV light with a quantum yield of 26%. Compared to the compound [Eu(dbm)$_3$(H$_2$O)$_2$], the photoluminescence intensity of Eu@glass is dramatically enhanced, and the emission lifetime of Eu^{III} (0.517 ms) more than an order of magnitude longer.

Key words: europium; luminescence; film; sol-gel

Introduction

The photoluminescent properties of lanthanoid compounds have drawn extensive attention due to their attractive features, such as characteristic sharp-line emissions, long emission lifetimes and high quantum yields [1-4]. These features make *Ln*^{III}-based materials suitable for use in a wide range of applications, such as sensing [5-11], imaging [12, 13], and phosphors in lighting [14-18]. Due to the nature of the 4f-4f transition, the direct excitation of *Ln*^{III} is rather inefficient. In lanthanoid complexes, the photon energy can be absorbed efficiently by its surrounding organic ligands upon which energy is transferred to the *Ln*^{III} emitting center, a phenomenon that is known as the “antenna effect” [19, 20].

The rather poor mechanical properties of lanthanoid complexes limit their technological applications [21, 22]. One of the solutions is to introduce the lanthanoid complex into a polymer or silica-based matrix, which can be used to fabricate luminescent thin films [23-31]. Transparent luminescent thin films containing lanthanoid complexes have been demonstrated to be useful for application in agriculture and horticulture [32, 33], lighting [34], Si-based solar cells [35] and as luminescent solar concentrators (LSCs) [21, 36].

The purpose of the research reported in this paper was to develop a new ligand that is able to bind europium and create a thin layer on glass surfaces. Such material potentially can been applied as luminescent solar concentrator or in Si-based solar cells. In this paper, we report the synthesis of the new silylated diamidopyridyl ligand **L** (scheme 1) and the corresponding Eu^{III} organic-inorganic silicate hybrid film. The thin film on the surface of glass has been obtained via a sol-gel process.

Experimental

Materials

All chemicals and solvents were purchased from commercial sources and used without further purification. The compound $[Eu(dbm)_3(H_2O)_2]$ (Hdbm = dibenzoylmethane) was synthesized following a literature procedure [37]. IR spectra were recorded on a Perkin Elmer UATR Two FT-IR spectrometer set to a resolution of 4 cm^{-1}. The solid-state excitation spectra were recorded using a Shimadzu RF-5301PC spectrofluoriphotometer. The solid-state emission spectra were recorded with an irradiation-calibrated CCD sensor (Avantes AvaSpec-2048UA). Solid-state absorption spectra were recorded with the Avantes absorbance setup, using the Avantes AvaSpec-2048UA CCD spectrometer as detector, and the AvaLight-DH-S Deuterium-Halogen light source. The fluorescence lifetime was measured using a Hamamatsu R928P photomultiplier connected to a Tektronix DPO4054 digital scope, a Carl Zeiss M20 grating monochromator was used to select the emission wavelength. A Labview program was used to collect and to average the data. Excitation was done with an Ekspla NTB342B Nd:YAG laser equipped with a OPO and SHG to generate 5ns pulses at a 10 Hz rate. The photoluminescence quantum yield was determined using the absolute method [38]. An integrating sphere (Avantes AvaSphere 30REFL) was connected to an irradiance-calibrated CCD spectrometer (Avantes AvaSpec-2048UA). A 1000 W Xenon lamp (LOT) and a Spex monochromator were used as the excitation source.

Ligand synthesis

Dimethyl pyridine-2,6-dicarboxylate (1)

Thionyl chloride (26.80 ml, 372 mmol) was added dropwise to methanol (125 ml) at 0 °C, then solid 2,6-pyridinedicarboxylic acid (10.00 g, 60 mmol) was added to this solution. The reaction mixture was stirred at room temperature for 18 h, and then refluxed for 2 h. After evaporation of the solvent, the residue was dissolved in diethyl ether (150 ml) and washed with water (3×30 ml). The organic layer was dried with anhydrous $MgSO_4$ and the solvent was evaporated in *vacuo*. The crude product was used in the next step reaction without further purification. Yield: 11.01 g, 94%. ^1H NMR (300 MHz, $CDCl_3$): $\delta = 8.33$ (d, J = 7.8 Hz, 2H, Ph), 8.05 (t, J = 7.8 Hz, 1H, Ph), 4.04 (s, 6H, CH_3) ppm. ^{13}C NMR (75 MHz, $CDCl_3$): δ =165.0, 148.2, 138.4, 128.0, 53.2 ppm.

N,N'-bis(2-aminoethyl)-2,6-pyridinedicarboxylic diamide (2)

Intermediate **2** was synthesized following a literature procedure.[39] 1,2-Diaminoethane (8.95 g, 149 mmol) was added to a solution of **1** (4.00 g, 20.4 mmol) in 200 ml methanol, and the mixture was stirred at room temperature for 12 h. After the reaction, the formed precipitate (oligomeric by-products) was removed by filtration and the filtrate was evaporated in *vacuo*. The residue was dissolved in 30 ml butanol and evaporated to dryness to further remove 1,2-diaminoethane. A yellowish solid was obtained, which was used in the next step without further purification. Yield: 4.84 g, 95%. ^{1}H NMR (300 MHz, MeOD): δ =8.31-8.14 (m, 3H, Ph), 3.55 (t, *J* = 6.3 Hz, 4H, CH_2), 2.93 (t, *J* = 6.3 Hz, 4H, CH_2) ppm. ^{13}C NMR (75 MHz, MeOD): δ =164.9, 148.8, 139.1, 124.4, 41.8, 40.8 ppm.

Ligand L.

To a solution of **2** (2.56 g, 10.2 mmol) in 200 ml toluene was added 3-triethoxysilanylpropyl isocyanate (5.54 g, 22.4 mmol), the mixture was refluxed for 24 h, after which the solvent was removed and the oil was kept at –20 °C. The resulting white precipitate was collected by filtration, washed with diethyl ether and dried in *vacuo*. Yield: 2.01 g, 26%. ^{1}H NMR (300 MHz, $CDCl_3$, Figure S1): δ =8.28 (d, *J* = 7.8 Hz, 2H, Ph), 8.00 (t, *J* = 7.5 Hz , 1H, Ph), 3.84 (q, *J* = 7.0 Hz, 12H, OCH_2Me), 3.69-6.60 (m, 4H, CH_2), 3.60-3.49 (m, 4H, CH_2), 3.16 (q, *J* = 6.7 Hz, 4H, CH_2), 1.69-1.60 (m, 4H, CH_2), 1.20 (t, *J* = 7.8 Hz, 18H, CH_3), 0.61 (t, *J* = 8.1 Hz, 4H, CH_2) ppm. ^{13}C NMR (75 MHz, $CDCl_3$, Figure S2): δ =164.4, 160.0, 148.6, 138.9, 124.2, 58.5, 43.1, 41.7, 39.8, 23.6, 18.4, 7.6 ppm. Selected IR data (*ν*): 3284 (m), 2968 (m), 2929 (m), 2886 (m), 1664 (m), 1637 (m), 1547 (s), 1497 (m), 1446 (m), 1389 (w), 1359 (w), 1291 (m), 1268 (m), 1249 (m), 1167 (m), 1100 (s), 1074 (vs), 1003 (w), 953 (m), 847 (w), 775 (m), 748 (m), 681 (m), 646 (m), 479 (w) cm^{-1}.

Preparation of luminescent hybrid material Si-Eu

The ligand **L** (6.7 µmol, 5.0 mg) and $[Eu(dbm)_3(H_2O)_2]$ (3.3 µmol, 2.8 mg) were dissolved in 5 ml of a mixture of $EtOH/H_2O$=9.5:0.5, and stirred at room temperature for 3 days. The white powder precipitate was collected by filtration, washed with acetone, and dried at 50 °C.

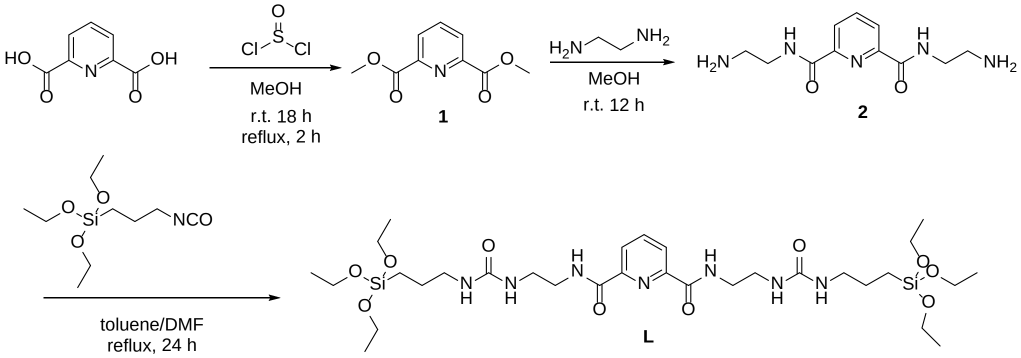
Preparation of luminescent silica film (Eu@glass)

Glass substrates (Corning® cover glass, 18×18 mm) were cleaned with acetone in an ultrasonic bath for 5 min, then dried in the oven. The substrates were immersed in a mixture of hydrogen peroxide and sulfuric acid, and then stored in distilled H_2O. $[Eu(dbm)_3(H_2O)_2]$ (3.3 µmol, 2.8 mg) was dissolved in a solution of ligand **L** (6.7 µmol, 5.0 mg) in 5 ml of a mixture of $EtOH/H_2O$=9.5:0.5, and the reaction mixture was stirred at room temperature for 30 min. A transparent thin film was prepared by spin-coating of two drops of this solution on the surface of glass substrates at spin rate of 3000 rpm. A non-transparent film was made by dropping the solution onto a glass substrate, which was then dried in air.

Results and discussion

Synthesis and characterization

The ligand **L** was synthesized following the procedure in Scheme 1 in an overall yield of 23%. The ligand was mixed into an ethanol-methanol solution containing the compound [Eu(dbm)$_3$(H$_2$O)$_2$], yielding a transparent solution that was either spin-coated or drop-casted onto a thoroughly cleaned and pre-treated glass plate. Drop-casting resulted in a non-transparent film, whereas spin-coating led to a clear and transparent film. In the description below the resulting films are indicated as Eu@glass.



Scheme 1. Synthesis route of the ligand **L**.

The FTIR spectra of the compound [Eu(dbm)$_3$(H$_2$O)$_2$], the ligand **L** and the powder Si-Eu are shown in Figure 1. The broad peak at 3351 cm^{-1} in the FTIR spectrum of [Eu(dbm)$_3$(H$_2$O)$_2$] can be assigned to the presence of the two water molecules (trace a, Figure 1), whereas the peaks at 1595 and 1454 cm^{-1} are assigned to the stretching vibration of the carbonyl groups of the dbm ligand [40, 41]. The absorbances in the range of 750-680 cm^{-1} are assigned to the C-H out-of-plane wagging of the phenyl rings in the dbm ligand [25, 42]. In the FTIR spectrum of the ligand **L**, the peaks at 1637 cm^{-1} (C=O stretching vibration) and 1547 cm^{-1} (N-H bending vibration) are assigned to the amide groups (trace b, Figure 1) [30]. The peaks at 1074 and 479 cm^{-1} belong to the stretching and bending vibration of the Si-O groups, respectively [43]. The broad peak at 3284 cm^{-1} can be assigned to the stretching vibration of the amide N-H. In addition to the NMR spectra this IR spectrum supports that the ligand **L** was synthesized successfully. In the FTIR spectrum of Si-Eu, a broad band centered at 1039 cm^{-1} (Si-O-Si asymmetric stretching vibration) indicates the successful formation of the Si-O-Si framework (trace c, figure 1) [22, 23, 44]. Furthermore, the two strong absorptions at 1661 and 1535 cm^{-1} assigned to the amide groups are slightly shifted compared to the spectrum of the ligand **L**, which might be ascribed to the coordination of ligand **L** to Eu^{III} via the pyridine nitrogen and the amide carbonyl oxygens [45].

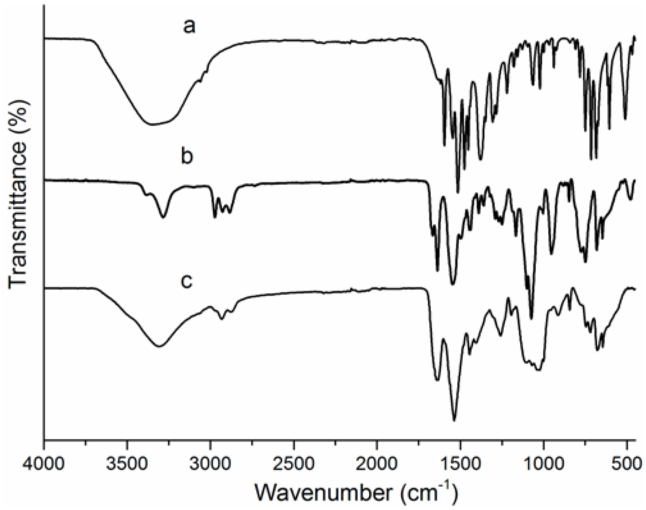


Figure 1. FTIR spectra of a) $[Eu(dbm)_3(H_2O)_2]$ b) ligand **L** and c) Si-Eu.

The solid-state absorption spectra of $[Eu(dbm)_3(H_2O)_2]$, Si-Eu and ligand **L** are shown in Figure 2. Only a narrow absorption peak with a maximum at 275 nm in is present in the absorption spectrum of ligand **L**. Compared to the broad absorption peak in the spectrum of $[Eu(dbm)_3(H_2O)_2]$, the spectrum of Si-Eu is narrower in the blue region. Furthermore, the hybrid material Si-Eu does not absorption at wavelengths higher than 500 nm, which means that any luminescent emission from the Eu^{III} center cannot be reabsorbed by Si-Eu; the negative peaks in the Si-Eu absorption spectrum are due to emission of the Eu^{III} center.

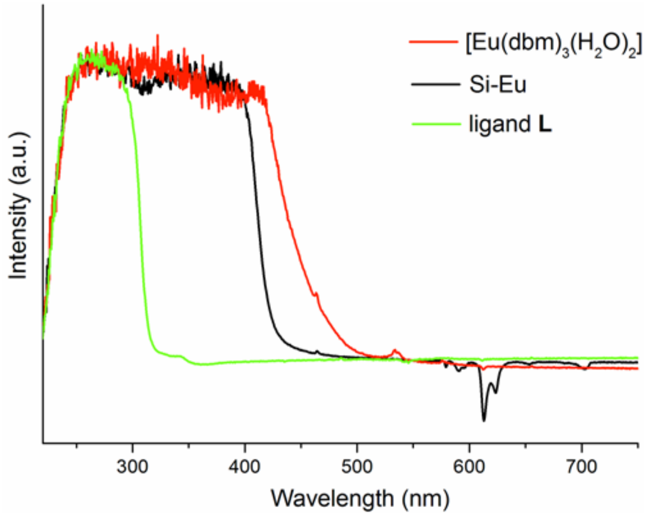


Figure 2. Solid state absorption spectra of $[Eu(dbm)_3(H_2O)_2]$, Si-Eu and ligand **L**.

Luminescent properties

Under the irradiation of a high-pressure Hg lamp (366 nm), [Eu(dbm)$_3$(H$_2$O)$_2$] shows very weak red emission (Figure 3a,d); the weak emission is ascribed to quenching of the high-energy state through coupling with the O–H oscillators of the coordinated water molecules [46-50]. In contrast, the drop-casted Eu@glass exhibits an intense red-color emission (Figure 3e). The transparent film Eu@glass prepared by spin-coating only exhibits red-color emission at the borders of the glass plate due to total internal reflection [51]. (Figure 3f).

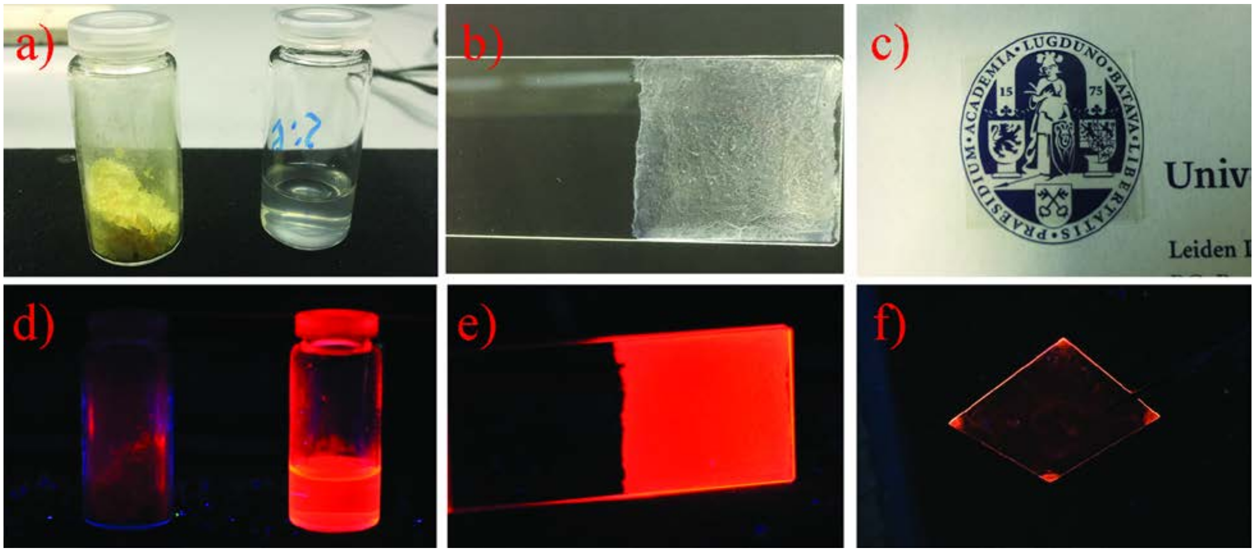


Figure 3. Pictures of [Eu(dbm)$_3$(H$_2$O)$_2$] (yellow solid in vial) and the hybrid gel (transparent liquid in vial; a and d); Eu@glass on a glass substrate obtained by drop-casting (b and e); Eu@glass on glass substrate obtained by spin coating (c and f). a, b and c: ambient light; d, e and f: 366 nm UV light. In figure f), the red emission is concentrated to the border of glass due to total internal reflection [51].

The excitation and emission spectra of the film Eu@glass have been recorded (Figure 4). Upon excitation with 385 nm UV light the emission spectrum of Eu@glass shows the five emission peaks that are characteristic for the Eu^{III} ion at 578, 590, 615, 652 and 699 nm, arising from the $^5D_0 \rightarrow {}^7F_J$ (J = 0-4) transitions. No phosphorescence emission from the ligand is observed, indicating an efficient energy-transfer process from the excited state of the ligand to the Eu^{III} ion. The emission peak at 590 nm ($^5D_0 \rightarrow {}^7F_1$) is a magnetic dipole (MD) transition and its intensity is insensitive to the coordination environment. However, the $^5D_0 \rightarrow {}^7F_2$ transition at 615 nm is a forced electric dipole (ED) transition and its intensity is sensitive to the Eu^{III} surroundings [52]. The emission spectrum is dominated by the $^5D_0 \rightarrow {}^7F_2$ transition at 615 nm and the emission intensity of the $^5D_0 \rightarrow {}^7F_1$ transition is rather weak, indicating lack of an inversion center at the Eu^{III} site [53]. Compared to [Eu(dbm)$_3$(H$_2$O)$_2$], the significant luminescence enhancement of Eu@glass gives a strong indication that the ligand **L** is coordinated to the Eu^{III} emitting center, having replaced the water molecules. The emission quantum yield of the Eu@glass has been measured using the absolute method and was calculated to be 26% [38]. The emission of [Eu(dbm)$_3$(H$_2$O)$_2$] is too weak for quantum yield determination in our setup, but has been reported to be 1% [49].

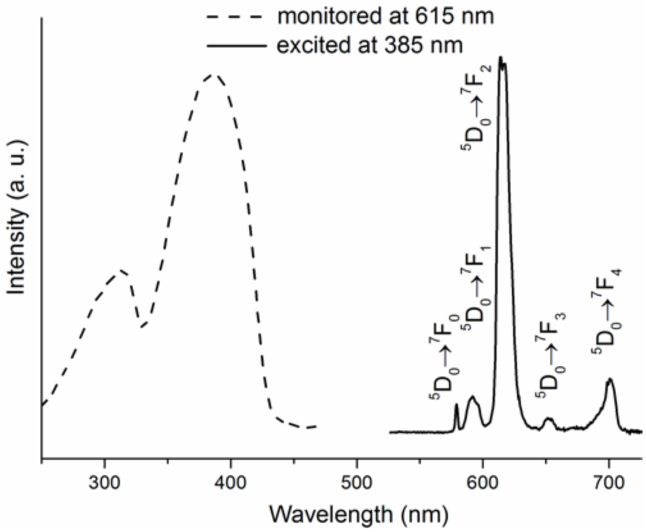


Figure 4. Excitation (left) and emission (right) spectra of Eu@glass.

The $^5D_0$ decay curves monitored at 615 nm for the Eu@glass and the compound [Eu(dbm)$_3$(H$_2$O)$_2$] are shown in Figure 5. Both decay curves display mono-exponential behavior. The emission lifetime of the Eu^{III} ion in [Eu(dbm)$_3$(H$_2$O)$_2$] is 0.048 ms, whereas the lifetime of the Eu^{III} emission in Eu@glass is 0.517 ms. The shorter emission lifetime of the Eu^{III} ion in [Eu(dbm)$_3$(H$_2$O)$_2$] is due to the quenching of the emission by the coordinated water molecules. The dramatically longer lifetime of emission in the Eu@glass further demonstrates that the ligand **L** is coordinated to the Eu^{III} ion.

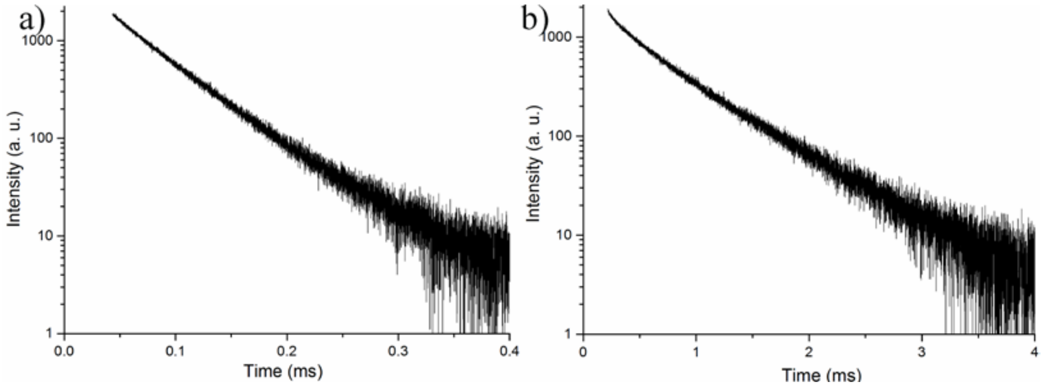


Figure 5. The decay curves of a) [Eu(dbm)$_3$(H$_2$O)$_2$]; b) Eu@glass monitored at 615 nm. (λ_{ex}=355 nm).

Conclusion

The luminescent thin film, Eu@glass, has been prepared on a glass surface via a sol-gel method using a silylated ligand **L** and [Eu(dbm)$_3$(H$_2$O)$_2$]. The film exhibits bright red luminescence under the excitation of near UV light. The considerable enhanced emission intensity and longer lifetime of the Eu@glass film confirm the coordination of the ligand **L** to the Eu^{III} ion, as it indicates the reduction of the quenching effect by coordinated water molecules.

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Appendix A. Supplementary data

Spectroscopic details in the supplementary data include: ^{1}H NMR and ^{13}C NMR spectra of ligand **L**.

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