Cytotoxic Effect of Samarium(III)-Based Complexes: Synthesis, Characterization and Photoluminescence Properties

AHMET ORAL SARIOGLU (✉ aoralsarioglu@gmail.com)

Gaziantep University: Gaziantep Universitesi <https://orcid.org/0000-0001-7787-7968>

demet taşdemir kahraman

Gaziantep Universitesi Tıp Fakültesi

Aysegül köse

Kahramanmaraş İstiklal Üniversitesi

mehmet sönmez

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Abstract

Three new homoleptic six-coordinate Sm(III) complexes have been synthesized from diketone ligand with different substituent groups. The structural properties of these compounds have been investigated using elemental analysis, UV-Vis, and FT-IR approaches. Then, their photoluminescence properties of the synthesized Sm complexes have been examined.

Also, the cytotoxic activities of the complexes have been studied against to human epidermoid carcinoma cell (HTB-54) and normal bronchus epithelial cell (BEAS-2B). According to result, the $[\text{Sm}(\text{MPBP-}^{1,3}\text{PPon})_3 \cdot \text{H}_2\text{O}]$ (IC_{50} : 11.05 μM) complex was more active than $[\text{Sm}(\text{BMP-}^{1,3}\text{PPon})_3 \cdot 2\text{H}_2\text{O}]$ (IC_{50} : 58.64 μM) and $[\text{Sm}(\text{BCP-}^{1,3}\text{PPon})_3 \cdot \text{H}_2\text{O}]$ (IC_{50} : >500 μM) complexes. Otherwise it was no effect on normal cell (BEAS-2B). This result shows that complex $[\text{Sm}(\text{MPBP-}^{1,3}\text{PPon})_3 \cdot \text{H}_2\text{O}]$ has the potential to be the ideal chemotherapeutic drug.

Introduction

The luminescence characteristics of rare earth metal complexes with various organic ligands have recently received a lot of attention. There are many studies in the literature on the synthesis and photophysical characteristics of lanthanide complexes with different substituents of β -diketone ligands. [1].

The lanthanide complexes have different applications such as light-emitting diodes (LEDs) [2–4], lasers [5–8], sensors [9–11], luminescent probes in biomedical [12–16], solar energy conversion [17, 18], single molecule magnets [19–24], superconducting materials [25–30] etc.

Ln(III) ions show distinct photophysical features, including intra-configurational 4f–4f transitions, narrow emission bands, a significant Stoke's shift, a long luminescence decay time, and strong luminescence generated by electron transitions between long-time emission spectra. [31–33].

The lanthanide ions are one of the essential components because of their spectroscopic and magnetic properties in the preparation of new materials and as suitable probes in studies of biological systems. [34].

Lung cancer is described as the deadliest type of cancer that causes 1.76 million deaths out of 2.09 million cases in the World [35]. There are two commonly known groups of lung cancers: small cell and non-small cell lung cancer. Epidermoid cancer, also called squamous cancer, is a type of non-small cell lung cancer [36]. Non-small cell lung cancer (NSCLC) is the most common group of lung cancer, occurring in eight or nine out of ten cases. Despite the use of chemotherapy, radiotherapy and immunotherapy, the 5-year survival rate is 15% when all cases are included. Unfortunately, the chemotherapy drugs such as cisplatin-based cause side effects on renal [37], liver [38] cells after treatment. For years, especially pharmacology and chemist researchers have been synthesized hundreds of new potential anti-cancer compounds to reduce the toxic effect.

We present the synthesis, photophysical characteristics and cytotoxic impact of new Sm(III) complexes in this study. The Sm(III) complexes were synthesized from the reaction of 1-(4-methoxyphenyl)-3-(4-tert-butylphenyl)-1,3-propanedione (MPBP-^{1,3}PPon), 1,3-bis(4-methoxyphenyl)-1,3-propanedione (BMP-^{1,3}PPon), 1,3-bis(4-chlorophenyl)-1,3-propanedione (BCP-^{1,3}PPon). The photoluminescence features of Sm(III) complexes were examined in DMF. On the other hand, we evaluated anti-cancer effects of [Sm(MPBP-^{1,3}PPon)₃·H₂O], [Sm(BMP-^{1,3}PPon)₃·2H₂O] and [Sm(BCP-^{1,3}PPon)₃·H₂O] complexes on epidermoid cancer cell lines (HTB-54).

Experimentel

Materials and instruments

All the materials were acquired with a stated purity of 99% from Sigma-Aldrich Chemical and Merck Company and utilized without additional purification. On a PG Instruments T80 + UV/Vis spectrometer, the UV-Vis spectrums were recorded from a 1x10⁻³ M solution of the compounds in DMF, DMSO and DCM from 800 to 190 nm. The IR ATR spectrum was produced using the Attenuated Total Reflectance (ATR) model on a Perkin Elmer FT-IR spectrometer between 4000 - 400 cm⁻¹ at spectral intervals of 2 cm⁻¹. Elemental analyses of the substances were performed using a Thermo Scientific Flash 2000 CHNS analyzer. The compounds excitation/emission spectra were measured with a Perkin Elmer LS55 luminescence spectrometer.

Preparation of Sm(III) complexes

A solution of MPBP-^{1,3}PPon (232 mg, 0.75 mmol), BMP-^{1,3}PPon (213 mg, 0.75 mmol), BCP-^{1,3}PPon (220 mg, 0.75 mmol) in dry ethanol (25 ml) was added dropwise into a solution of SmCl₃ (64 mg, 0,25 mmol) in absolute ethanol (10 ml) under stirring at 75 °C for 5h. Then, the pH of the reaction mixture was adjusted to around 6 using NaOH (aq). The yellow powder was filtered off, washed with ethanol and dried in vacuo for 48 hours.

[Sm(MPBP-^{1,3}PPon)₃·H₂O (Fig. S1): Yield: 45%, mp: 245-248 °C, UV vis (DMF) λ_{max} nm (log ε): 360 (1.697), 268 (0.825). IR v ATR(cm⁻¹): 3044 (br,C—H arm); 2956 (aliphaticcarbon); 1738 (str, C=O); 1595 (m, enolic C=C); 1251 (methoxy C-O-C); 504 (Sm—O). Chemical Formula: C₆₀H₆₅O₁₀Sm; Molecular Weight: 1186,58; Anal.Calcd.For C₆₀H₆₅O₁₀Sm (%): C, 65.72; H, 5.97. Found (%): C, 65.41; H, 5.65.

[Sm(BMP-^{1,3}PPon)₃·2H₂O (Fig. S2): Yield: 77%, mp: 238-241 °C, UV vis (DMF) λ_{max} nm (log ε): 365 (2.102), 268 (0.959). IR v ATR(cm⁻¹): 3066 (br,C—H arm); 2958 (aliphaticcarbon); 1738 (str, C=O); 1597 (m, enolic C=C); 1255 (methoxy C-O-C); 465 (Sm—O). Chemical Formula: C₅₁H₄₉O₁₄Sm; Molecular Weight: 1036,28; Anal.Calcd.For C₅₁H₄₉O₁₄Sm (%): C, 59.11; H, 4.77. Found (%): C, 59.06; H, 4.34.

[Sm(BCP-^{1,3}PPon)₃].H₂O (Fig. S3): Yield: 41%, mp: 261-264 °C, UV vis (DMF) λ_{max} nm (log ϵ): 358 (1.505), 268 (0.994). IR v ATR(cm⁻¹): 3069 (br,C—H arm); 2911 (aliphaticcarbon); 1737 (str, C=O); 1587 (m, enolic C=C); 483 (Sm—O). Chemical Formula: C₄₅H₂₉Cl₆O₇Sm; Molecular Weight: 1044,79; Anal.Calcd.For C₄₅H₂₉Cl₆Sm (%): C, 51.73; H, 2.80. Found (%): C, 51.54; H, 2.71.

Cytotoxic Result:

The effects of [Sm(MPBP-^{1,3}PPon)₃].H₂O) and [Sm(BMP-^{1,3}PPon)₃].2H₂O on all concentrations (31, 62, 125, 250, 500 μ M) were statistically significant when compared with DMSO in HTB-54 cells (p<0.001). [Sm(BCP-^{1,3}PPon)₃].H₂O complexes has no effect on cancer cells. For these reason, we were not studied on BEAS-2B for toxic experiment. Inhibitory concentrations of [Sm(MPBP-^{1,3}PPon)₃].H₂O) was determined as 11.05 μ M in HTB-54 cells. Similarly [Sm(BMP-^{1,3}PPon)₃].2H₂O was 58.64 μ M. So [Sm(MPBP-^{1,3}PPon)₃].H₂O) complex was better anti-cancer effect than [Sm(BMP-^{1,3}PPon)₃].2H₂O for cancer cells (**Figure 2**). In addition, [Sm(MPBP-^{1,3}PPon)₃].H₂O) complex has no toxic effect on healthy cell (BEAS-2B) so this compound is more suitable for ideal drug properties and has potential therapeutic agent (**Figure 3**).

IR spectra

Within the range of 4000–400 cm⁻¹, the IR spectra of compounds were determined. The IR spectrums of the [Sm(MPBP-^{1,3}PPon)₃].H₂O (**Fig. S4**), [Sm(BMP-^{1,3}PPon)₃].2H₂O (**Fig. S5**) and [Sm(BCP-^{1,3}PPon)₃].H₂O (**Fig. S6**) complexes showed a weak broad absorption bands at 3044, 3066 and 3069 cm⁻¹ assigned to the aromatic carbon ν (C^{3/4}H) a stretching vibration [40], absorption bands at 2956, 2958 and 2911 cm⁻¹ assigned to the aliphatic carbon ν (C^{3/4}H) a stretching vibration [41], respectively. The strong characteristic vibration bands of the lanthanide complexes occurring at 1738 and 1737 cm⁻¹ are due to carbonyl ν (C=O) [42,43]. Also, the absorption bands observed at 1595, 1597 and 1587 cm⁻¹ belong to the enolic carbon ν (C=C) a stretching vibration [44] and absorption bands at 1251 and 1255 cm⁻¹ belong to the methoxy groups ν (C^{3/4}O^{3/4}C) a stretching vibration, respectively. The results show that ligands can effectively coordinate with the Sm(III) ion.

3.3. UV-Vis spectra

The UV-Vis spectra of Sm(III) complexes were obtained in the range of 190-1100 nm by using DMF, DMSO and DCM solvents and the corresponding spectra are presented in **Figure 4-6**. These bands found in the UV-Vis spectra of Sm(III) complexes at 360, 365, and 358 nm in the DMF solution are linked to the singlet-singlet n- π^* transition of enol groups. Furthermore, the π - π^* transition of phenyl rings and carbonyl groups is ascribed to the absorption maxima at 268 nm. In DMSO, DMF, and DCM, the absorption wavelength of Sm(III) complexes was found to be slightly red shifted depending on the polarity of the solvent. [45-48].

Photoluminescence properties

The Sm(III) complexes of β -diketone derivatives showed a strong absorption band in the range of 300–410 nm due to the π - π^* electronic transition of the conjugated β -diketone ligands. Therefore, we investigated the emission properties of the complexes in DMF (10^{-5} M) solution upon excitation at the maximum π - π^* transition. The photoluminescence data are tabulated in **Table 1**. Emission spectra of [Sm(MPBP-^{1,3}PPon)₃] $\cdot$ H₂O), [Sm(BMP-^{1,3}PPon)₃] $\cdot$ 2H₂O and [Sm(BCP-^{1,3}PPon)₃] $\cdot$ H₂O are given in **Figure 7**. The ligands in the complexes [Sm(MPBP-^{1,3}PPon)₃] $\cdot$ H₂O), [Sm(BMP-^{1,3}PPon)₃] $\cdot$ 2H₂O and [Sm(BCP-^{1,3}PPon)₃] $\cdot$ H₂O are similar differing in the substitute groups on the pheny rings. The substitute groups did not cause considerable shifts in the excitation spectra of the complexes. In DMF, the complexes showed several emission bands in the range of 400–810 nm. All three complexes exhibited strong emission band in the range of 350–550 nm due to the ligand based emission $^1S_1 \rightarrow ^1S_0$. The substitute groups affect the ligand based emission maximums as well as intensities. The chloride groups has caused a considerable blue shift yet showed the highest emission intensity. This was observed for similar complexes reported by our group [40]. Complex [Sm(MPBP-^{1,3}PPon)₃] $\cdot$ H₂O) shows Sm(III) based f-f transitions at 578, 586, 634, 729, 768 and 824 nm and these emission bands were assigned to f-f transition $^4G_{5/2} \rightarrow ^6H_{5/2}$ (forbidden transition), $^4G_{5/2} \rightarrow ^6H_{7/2}$ (magnetic dipole transition), $^4G_{5/2} \rightarrow ^6H_{9/2}$ (electric-dipole transition), $^4G_{5/2} \rightarrow ^6H^{11/2}$ (forbidden transition). The intensive emission band observed for electric-dipole transition ($^4G_{5/2} \rightarrow ^6H_{9/2}$) are associated with the coordination structures related to odd parity [49]. The coordination numbers and geometry around Sm(III) centre in those complexes are expected to be similar and the emission spectra due to the electric-dipole transition ($^4G_{5/2} \rightarrow ^6H_{9/2}$) are almost identical in all complexes. In the spectra of [Sm(BCP-^{1,3}PPon)₃] $\cdot$ H₂O, $^4G_{5/2} \rightarrow ^6H_{9/2}$ (electric-dipole transition) and $^4G_{5/2} \rightarrow ^6H^{11/2}$ (forbidden transition) transitions were observed at 638 and 761 nm, respectively. The other f-f transitions were not observed.

Table 1. Photoluminescence data for the compounds.

Compound	Photoluminescence	
	Exc.	Em.
[Sm(MPBP- ^{1,3} PPon) ₃] $\cdot$ H ₂ O)	308	426, 578, 586, 634, 729, 768, 824
[Sm(BMP- ^{1,3} PPon) ₃] $\cdot$ 2H ₂ O	310	424, 581, 639, 729, 756, 816
[Sm(BCP- ^{1,3} PPon) ₃] $\cdot$ H ₂ O	310	408, 638, 761

Results And Discussion

Cytotoxic assay

Human epidermoid carcinoma cell (HTB-54) and normal bronchus epithelial cells (BEAS-2B) were cultured with different concentrations of the three complexes. The MTT test was used to determine cell viability. According to optical density results we calculated half maximal inhibitory concentration (IC_{50}).

Cell Culture

HTB-54 and BEAS-2B cells were grown 10% fetal bovine serum (FBS; Gibco, USA) and %1 antibiotic (Gibco, USA) supplemented in RPMI at 37°C, %5 CO₂ conditions.

Chemical Exposure

Cells were cultivated in 96 well plates (2000 cells/ml) for 48 h. The culture medium contained 10% FBS and % 1 antibiotic in RPMI medium. When cell proliferation % 70 in cell culture plates, medium was changed with serum free RPMI for 16 hours. The cells were exposed to various concentrations (0, 31, 62, 125, 250, 500 μ M) of complexes with serum free medium for 24h. The untreated cells were used as control [39].

MTT Assay (Cell Viability)

According to MTT assay; Cell culture media replaced with 100 μ l MTT (Gibco, USA) solution (mg/ml) to each well. The culture was incubated for 1h until living cell stained. We checked with microscope and discarded MTT solution. We added 100 μ l dimethyl sulfoxide (DMSO) and measured at 570 nm by a plate reader (Biotek, USA) [39].

Statistical analysis: MTT results were analyzed for non-normally distributed Mann-Whitney U test. We have analyzed treatment groups compared to the control group (DMSO including to high concentration). According to finding data, p values < 0.05 were noted to be significant. Prism 6 program used for statistical analysis (GraphPad Software, Inc, San Diego, USA).

Conclusion

A new series of ternary samarium (III) complexes with diketone structures including various groups has been successfully synthesized and characterized. The complexes showed the ligand-based emission $^1S_1 \rightarrow ^1S_0$ in the range of 350-550 nm. The β -diketone ligands caused an antenna effect and complexes exhibited Sm(III) based f-f transitions. The HTB-54 and BEAS-2B cell lines were used to test their antiproliferative activities in vitro. According to results, $[Sm(MPBP-^{1,3}PPon)_3] \cdot H_2O$ (IC_{50} : 11.05 μ M) and $[Sm(BMP-^{1,3}PPon)_3] \cdot 2H_2O$ compounds stronger activity against the HTB-54 cancer cell line. But only one of these compounds shows the potential drug, which is compound $[Sm(MPBP-^{1,3}PPon)_3] \cdot H_2O$. Because compound $[Sm(MPBP-^{1,3}PPon)_3] \cdot H_2O$ has an inhibitory effect on the cancer cell HTB-54, while it is not effective on the healthy cell BEAS2B.

Declarations

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Author contributions Methodology, data curation, analyses and synthesis, AOS; anticancer study DTK; Photoluminescence study, AK; Conceptualization and supervision, MS.

Compliance with ethical standards

Conflict of interest The authors declare no competing interests.

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Figures

Figure 1

Synthetic scheme of the Samarium complexes

Figure 2

Cell viability of complexes: Effect of complexes against HTB54 cell line

Figure 3

Cell viability of complexes: Effect of complexes against BEAS-2B cell line

Figure 4

UV-Vis spectra of [Sm(MPBP-1,3PPon)3]·H₂O in DCM, DMF, DMSO.

Figure 5

UV-Vis spectra of $[\text{Sm}(\text{BMP-}^{1,3}\text{PPon})_3] \cdot 2\text{H}_2\text{O}$ in DCM, DMF, DMSO.

Figure 6

UV-Vis spectra of $[\text{Sm}(\text{BCP-}^{1,3}\text{PPon})_3] \cdot \text{H}_2\text{O}$ in DCM, DMF, DMSO.

Figure 7

Emission spectra of the complexes in DMF (10^{-5} M) (λ_{exc} : 308 for $[\text{Sm}(\text{MPBP-}^{1,3}\text{PPon})_3]\cdot\text{H}_2\text{O}$), 310 for $[\text{Sm}(\text{BMP-}^{1,3}\text{PPon})_3]\cdot 2\text{H}_2\text{O}$ and $[\text{Sm}(\text{BCP-}^{1,3}\text{PPon})_3]\cdot\text{H}_2\text{O}$)

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