



A Review on Luminescent Rare Earth Complexes

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Authors' contributions

This work was carried out in collaboration among all authors. Author YZ designed the study. All authors contributed in practical work and managed the analysis of the study. Author QS wrote the first draft of the manuscript. All authors read and approved the final manuscript.

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ABSTRACT

At present, rare earth elements are widely used in various industries. In this paper, luminescent rare earth complexes are taken as a starting point to explore the luminescence principles of several important rare earth elements. The commonly used synthesis methods of luminescent rare earth complexes in recent year are also summarized, mainly including co-precipitation method, high temperature solid phase method, sol-gel method and hydrothermal synthesis method. And prospects for the future research on luminescent rare earth complexes are made.

Keywords: Rare earth complexes; luminescence materials; synthesis method; light emitting diodes.

1. INTRODUCTION

Rare earth elements are widely used as luminescent materials. Their good optical, electrical, magnetic, and thermal properties depend on the arrangement of their electronic shells. Therefore, they are also widely used in

military, electronic and new materials fields, and is a hot area of academic discussion [1]. Luminescent rare earth complexes have many advantages, such as strong absorption of light, high conversion efficiency, wide range of emission wavelength, wide range of fluorescence lifetime, stable physical and chemical properties,

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high temperature and strong light resistance, etc. It has more excellent performance than other luminescent materials [2].

In the field of luminescent rare earth complexes, a relatively complete industrial chain has been formed in China. In recent years, the market for various types of luminescent rare earth complex materials demand has risen sharply, which has great potential for production and consumption. Light-emitting diodes (LEDs) are favored by a wide range of manufacturers because of their advantages of long use life, low power consumption, and environmental friendliness [3]. White light is the most common and common light source in life, and the study of luminescent rare earth complexes is the key to white light LEDs. Therefore, the research on the synthesis of luminescent rare earth complexes and the application of white light LEDs has theoretical significance and practical application value.

2. RESEARCH STATUS

In the past ten years, the number of patents applied for in the field of rare earth luminescent materials in China has ranked first in the world. China is a major producer and user of rare earths. The production of products based on rare earths accounts for about 90% of the world's total production. Among them, the production of many functional materials even reaches more than 70% of the world's total production [4].

In the field of lighting, China has made a major contribution to the production of rare earth luminescent complexes. According to relevant materials, the production of phosphors in China used to reach 25,000 tons per year, and our country provides more than half of energy-saving lamps and computers for the world [5]. At present, the products based on rare earth luminescence complexes are mostly produced in China, and the enterprises and factories of producing rare earth luminescence complexes are mainly located in the coastal areas, Gansu province and Shaanxi province [6].

In the face of the growing market demand and the need for higher quality, researchers are now trying to combine organic ligands with strong light absorption with rare earth central ions to better improve the luminescent properties of rare earth ions. Because suitable organic ligands have the advantage of transferring their energy to the rare earth central ion through a non-radioactive transition pathway, thus enabling better

sensitization of the rare-earth ion for luminescence [7].

At present, although there is no lack of market demand for lighting white LEDs, there are still obvious shortcomings in this field: the products emit harmful blue light in the process of use; the light source has a short service life; the price of luminescent materials in the overall product is relatively low, so people do not pay enough attention to it [8]; at the same time, the related technology of encapsulating the prepared rare earth luminescent complexes into the products and the exploration of the use performance of the products are incomplete.

Based on the importance of rare earth, it is called "the strategic element of the 21st century" and is an indispensable link in the process of preparing high-tech materials. Rare earth luminescent complexes are not only widely used in industrial production and promote the development of industrial production, they are also commonly used in people's life and improve people's quality of life. China needs to make good use of the trump card of rare earth resources to narrow the gap in resource utilization between China and other countries [9]. Therefore, the synthesis and research of new luminescent rare earth complexes are of great significance to further enhance the research and development in the field of rare earths in China.

At present, the main object of research is still the research and industrialization of rare earth luminescent materials for white LEDs. There are many technologies to realize white LEDs, among which fluorescence conversion white LEDs are the focus of current research. Related scientific researchers are creating more advantageous white LEDs, such as low color temperature and high power [10].

3. LUMINESCENCE MECHANISM of Eu^{3+} , Tb^{3+} AND Dy^{3+}

The most studied and practically valuable rare earth complexes are Eu^{3+} , Tb^{3+} and Dy^{3+} , so we focus on these three important rare earth elements for introduction. The luminescence process of rare earth complexes is a process that produces radiation when the rare earth complexes are subjected to the action of, for example, ultraviolet light, friction, rays or other excitation methods [11].

The reason why rare earth complexes have multiple luminescence types is due to the unique 4f electronic configuration and transition of rare earth ions. Although the 4f electrons are less affected by the outside world because of the shielding effect, they have abundant electronic energy levels due to their relatively large spin coupling constants which can cause J-energy level splitting [12].

3.1 Luminescence Mechanism of Eu^{3+}

Among rare earth luminescent materials, Eu^{3+} is the more commonly used activator for red luminescent materials.

Chunfang Guo [13] mentioned the fluorescence luminescence mechanism of rare earth europium complexes in their paper on the progress of luminescent materials of rare earth europium complexes. The fluorescence of rare earth europium complexes mainly emits light by the sensitization luminescence method. Through the energy transfer from excited ligands, the central ion receives energy and emits red fluorescence. This is called the "antenna effect" [14]. As shown in Fig. 1, the ligand absorbs UV light firstly and then transitions from the ground state S_0 to the lowest excited singlet state S_1 . The least excited singlet state crosses to the excited triplet state T_1 by inter-system scurry. The energy is transferred from the lowest excited triplet state to the vibrational state of europium ion by the vibrational coupling of the bond, and the ground state electron of europium ion is excited to the excited state. The characteristic fluorescence of Eu^{3+} is emitted when the electron returns to the

ground state from the excited state energy level.

It is worth noting that Eu^{3+} is used in blue phosphors, and its ground state electronic configuration is $4f^7 5d^0$. The energy level of 4f orbital is octet degenerated and produces d-f transition luminescence, which has the advantages of short excited state lifetime to nanosecond and adjustable spectrum [15,16].

3.2 Luminescence Mechanism of Tb^{3+}

Tb^{3+} is one of the commonly used activators in green rare earth luminescent materials.

Lu Tang mentioned in their paper on the preparation and luminescence properties of $\text{YVO}_4:\text{Eu}^{3+}$ and $\text{YVO}_4:\text{Tb}^{3+}$ nanoparticles that the luminescence of Tb^{3+} was mainly due to the light emitted by its 4f electrons jumping from the high energy levels of $^5\text{D}_4$ and $^5\text{D}_3$ back to the low energy level of $^7\text{F}_J$ ($J=0-6$). The blue emission at 416-418 nm and 430-450 nm correspond to the $^5\text{D}_3 \rightarrow ^7\text{F}_5$ and $^5\text{D}_3 \rightarrow ^7\text{F}_4$ transitions of Tb^{3+} , respectively. The green emission at 470-490 nm and 542-550 nm correspond to the $^5\text{D}_4 \rightarrow ^7\text{F}_6$ and $^5\text{D}_4 \rightarrow ^7\text{F}_5$ transitions of Tb^{3+} , respectively. When the concentration of incorporated Tb^{3+} is large, the green light emission produced by the $^5\text{D}_4 \rightarrow ^7\text{F}_5$ transition is the most significant [17].

3.3 Luminescence Mechanism of Dy^{3+}

The rare earth element ion Dy^{3+} is often used as an activator for long afterglow materials, but the mechanism of Dy^{3+} activation of persistent phosphor luminescence is still unclear [18].

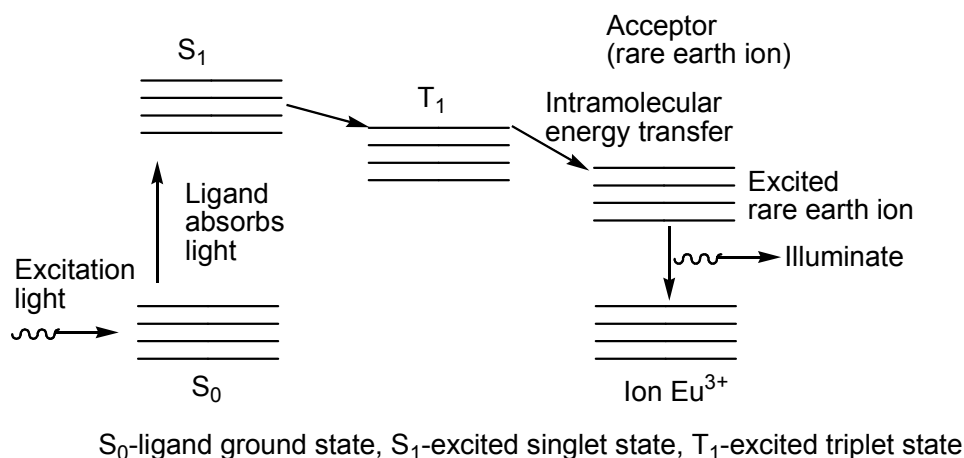


Fig. 1. Schematic diagram of antenna effect

T Matsuzawa suggested that in $\text{SrAl}_2\text{O}_4:\text{Eu}^{2+}:\text{Dy}^{3+}$, Dy^{3+} was the hole capture center, or called a hole trap. When the material was excited by UV and visible light, Eu^{2+} became Eu^+ . This process would leave a hole in the valence band, and the hole in the valence band would be transferred to the vicinity of Dy^{3+} and be captured by it. When the excitation was stopped, the holes captured by Dy^{3+} were released to the valence band by thermal excitation, and the holes were captured by Eu^+ to return to Eu^{2+} . So the electrons were recombined with the holes, and Eu^{2+} excited state electrons leaped to the ground state, producing long afterglow luminescence [19].

4. SYNTHESIS AND PREPARATION OF RARE EARTH COMPLEXES

The luminescent properties of rare earth complexes are related to the composition and structure of the complexes. Different synthesis methods have an impact on the properties and luminescence properties of the rare earth complexes [20]. Therefore, according to different needs, a suitable synthesis method should be selected. At present, the synthesis methods of rare earth complexes mainly include co-precipitation method, sol-gel method, hydrothermal synthesis method, microwave synthesis method, high temperature solid phase method, etc.

4.1 Co-precipitation Method

The co-precipitation method is a preparation method in which multiple cations in a solution are precipitated to generate a precipitation mixture or solid solution precursor.

Dong Zhu [21] successfully prepared phosphors of BaY_4O_9 system by using ammonium bicarbonate co-precipitation method and doped with different elemental ions, which could emit excellent orange-red light. Yang Liu [22] used CaF_2 ceramics as the matrix and rare earth ions as doping ions to prepare CaF_2 nanopowders by co-precipitation method to improve the optical quality. Akca et al [23] prepared $\text{Zn}_2\text{B}_2\text{O}_4$ phosphors using a wet chemical route, and the pure phase compounds were obtained by doping different concentrations of Pr. Chen et al. [24] synthesized $\text{Ce}_{0.33}\text{Zr}_{0.55}(\text{LaNdY})_{0.12}\text{O}_2$ by the sulfate assisted co-precipitation method, and enabled the formation of secondary loose accumulation of sub-particles, which resulted in

higher thermal stability of CZ-S samples during being aged at 60°C .

The co-precipitation method for synthesizing rare earth complexes not only allows for the refinement and homogeneous mixing of raw materials, but also has the advantages of simple process, low calcination temperature and short time, and good product performance.

However, in the process of co-precipitation, the addition of precipitating agent may cause the local concentration too high, resulting in agglomeration or the composition is not uniform enough, and this is something that needs to be solved.

4.2 High Temperature Solid Phase Method

The high temperature solid phase method is a traditional process. The compounds prepared by this method have the advantages of no agglomeration and good filling property, and it is still a commonly used method.

Nie et al. [25] prepared a novel apatite-type red light-emitting $\text{Ba}_2\text{La}_8(\text{GeO}_4)_6\text{O}_2:\text{Eu}$ phosphor by a high temperature solid phase method, which has a pure apatite phase without any impurities. Wang et al. [26] successfully synthesized $\text{Gd}_2\text{InSbO}_7:\text{Eu}^{3+}$ red phosphor through high temperature solid phase reaction, and this phosphor can be mixed with blue and green phosphors to make white LEDs. Jiuxu Xia [27] used the high temperature solid phase method to synthesize the $\text{Zn}_2\text{SiO}_4:\text{Eu}^{3+}$ system at a sintering temperature of 1350°C , and the luminescence properties of the $\text{Zn}_2\text{SiO}_4:\text{Eu}^{3+}$ system were studied.

However, the high temperature solid phase method needs to be carried out at high temperature ($1000\text{-}1500^\circ\text{C}$), which requires large energy consumption and has disadvantages such as coarse powder, low production efficiency, and easy to be mixed with impurities, so this synthesis method is mostly applied in industrial production that does not require fine preparation, and laboratory preparation is not commonly used.

4.3 Sol-gel Method

The sol-gel method refers to inorganic or metal alkoxides are polymerized and solidified through solution, sol and gel to form gel with space structure. After drying and heat treatment, nano-

particles and required materials are prepared. The luminescent rare earth complexes prepared by sol-gel method can improve the stability, high hardness, tensile strength, wear resistance and scratch resistance of the complexes [28].

Guo et al. [29] prepared cubic Gd_2O_3 nanocrystals doped with rare earth ions (Tm^{3+} , Er^{3+} and Yb^{3+}) by the sol-gel method. Lim et al. [30] also synthesized $CaGd_{2-x}(WO_4)_4:Er^{3+}/Yb^{3+}$ phosphor by the sol-gel method and improved its crystal structure, so that this synthetic route can be widely used in other similar complex compounds.

But this method has a long synthesis cycle and also suffers from products that are not easily controlled and prone to other reactions, especially oxidation.

4.4 Hydrothermal Synthesis Method

The hydrothermal method refers to a method in which water is used as a solvent under high temperature and high pressure in a sealed pressure vessel, then the reactants are dissolved and recrystallized to prepare the products.

Tang et al. [31] used sodium citrate as a chelating agent to prepare $NaY(MoO_4)_2:Pr^{3+}:Tb^{3+}$ phosphors by a hydrothermal method, which has a body-centered tetragonal structure, and the luminescent color of the phosphor would keep changing with temperature. Yu et al. [32] prepared white light $Ca_3(PO_4)_2:Dy^{3+}$ phosphors by hydrothermal synthesis, and investigated the effects of pH and calcination temperature on the morphology and luminescence properties of phosphors. Liuyang Yu et al. [33,34] synthesized a series of $NaGdF_4:Sm^{2+}/Dy^{3+}/Yb^{3+}/Er^{3+}/Zr^{4+}$ phosphors using a convenient hydrothermal method assisted by disodium ethylenediaminetetraacetate (EDTA-2Na), and achieved the emission of many different colors of light by changing the excitation wavelength and changing the doping amount of rare earth elements, etc. Hydrothermal method is a commonly used method for doping rare earth elements. Gnanam et al. [35,36] synthesized Eu-doped ceria and Dy-doped $CeVO_4$ nanorods by hydrothermal method.

Compared with the sol-gel and co-precipitation methods, hydrothermal synthesis method has the advantages of the crystalline powder being obtained without high temperature, the powder crystals being well developed, uniformly

distributed, small particles and light agglomeration.

4.5 Other Methods

In addition to the above-mentioned several solid-phase and liquid-phase synthesis methods, there are also gas-phase synthesis methods. Such as the gas-phase chemical thermal diffusion and permeation method [37], microwave synthesis method, spray pyrolysis method [38] and so on. These methods are developed with the development of science and technology, but currently, they are not widely used due to the limitations of the instruments and the harsh experimental conditions.

There is another method of the solid phase synthesis being called self-propagating synthesis. This method has a fast synthesis speed and relies on the exothermic reaction of the reaction itself, which can save resources and is a green synthesis method [39]. In recent years, the preparation of luminescent rare earth complexes by the impinging stream method has also become popular. This method can reduce the energy consumption of the reaction to a certain extent and improves the efficiency [40].

5. APPLICATIONS OF LUMINESCENT RARE-EARTH COMPLEXES

Because of the excellent performance of special optical, electrical, and thermal stability, luminescent rare earth complexes can be widely used in many fields such as manufacturing, industry, agriculture, biology and medicine and so on, which have economic value while promoting the rapid development of social high-tech industries.

Huang Li [41] introduced rare earth complexes into polymer matrices with specific functions in order to improve the thermal stability of rare earth complexes, and the synthesized carboxylic acid and amide rare earth metal complexes provided a pioneering idea for the industrialization of polymeric fluorescent materials. By changing the content of rare earth ions and doping materials, the fluorescence emission intensity is enhanced so that the prepared luminescent rare earth complexes can be applied in energy-saving, environment-friendly, green-use and long-life LED light-emitting diodes.

The luminescent rare earth complexes can be used in fluorescence flaw detection [42]. By irradiating with an ultraviolet lamp, rare earth complexes will emit light and convert ultraviolet rays into visible light, so that the human eye can see tiny cracks on the surface of the parts and troubleshoot in time.

The luminescent rare earth complexes are used in various displays or fluorescent screens. They can emit light at high temperatures while withstanding high current densities, restore the color fidelity and provide technical guarantees for the clarity and brightness of the screen.

Because of the high sensitivity and speed, the luminescent rare earth complex can be used to check the temperature distribution of different positions on the integrated circuit, and quickly and easily troubleshoot faults caused by overheating.

Solar fluorescent concentrators can be made using rare earth complexes [43], and the conversion of high energy photons into absorbed photons can increase the total light absorption of the cell and greatly improve the photoelectric conversion efficiency of the solar cell.

The photoluminescence properties of rare earths have been used in fluorescence immunoassays and rare earth fluorescent probes to sensitively probe the internal structure of molecules without direct contact and to contribute to the study of information on biological macromolecules.

Rare earth complex luminescent materials can be applied to agricultural films [44]. Lumogen [45] is usually used as a UV-enhanced material to convert UV light from sunlight into visible light that is beneficial to crop growth, and an efficient, stable and tunable UV-enhanced film promotes the photoenvironment and thermal environment required for plant photosynthesis to realize the increase of crop yield and income.

Guodong Qian et al. [46] found that the mixed dual rare earth organic complex luminescent materials had fluorescent properties in the temperature range of -263-25°C. Meanwhile, the fluorescence color changed with temperature in the temperature below 25°C. So this kind of luminescent materials is gradually applied in the field of low-temperature detection, which has strong application prospects and practical performance.

6. CONCLUSION AND OUTLOOK

At present, various scientific studies have shown that the luminescence properties of rare earth complexes are affected by many factors, mainly including the electronic layer structure of rare earth ions, the structure of the ligands, the type of central ions, the environment of the complexes, the synergistic reagent, and the energy transfer between ligands and rare earth ions.

Through the unremitting efforts of scientific researchers, the methods of improving the luminescent performance and efficiency of luminescent rare earth complexes have been greatly improved, and they have begun to show results in some fields [47]. However, the development of new luminescent rare earth complex materials [48], the improvement of the preparation process of luminescent rare earth complexes, and the search for ways to improve luminescent efficiency are still the focus of future research.

We can make efforts in the following areas: (1) Strengthening the study of the basic theories of rare earth materials, and only on the premise of familiarizing with the theories can we establish a theoretical system with guidance. (2) Looking for the opportunities for mass production, so that the materials produced in small and medium scale in the laboratory can be produced on a large scale and increase the possibility of their industrialization. (3) Enhancing the ability of independent innovation, combining rare earth complexes with novel and suitable substrates. (4) The development of rare earth luminescent materials should be mutually promoted and grown with the development of new materials and new application fields. The research on rare earth luminescent complexes can greatly promote the development of China's industry.

In order to make China's rare earth industry develop sustainably, it is necessary not only should we make innovations in research, but also need to pay close attention to the problem of unbalanced application of rare earth resources and pay attention to the development and protection of rare earth resources.

In summary, the research on rare earth luminescent complexes is multi-disciplinary and multi-directional, including physics, chemistry, device technology, and materials science [49]. We need to delve to study the preparation of rare

earth luminescent materials, the relationship between their luminescence mechanism, structure and properties in order to lay a certain foundation for the industrialization of rare earth luminescent complex materials [50]. The long-term and in-depth study of rare earth luminescent complexes will not only enrich the subject content of rare earth luminescent complexes, but also promote the development of the theory and practice of rare earth luminescent complexes.

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COMPETING INTERESTS

Authors have declared that no competing interests exist.

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