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Large-Size crystal based on rare earth-free Cu(I) hybrid trigger yellow light with high emissive quantum yields

Yuan-Lan Zhang*, Gui-Bin Wang

Co-Innovation Center for Sustainable Forestry in Southern China, Nanjing Forestry University, Nanjing, 210037, PR China

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ABSTRACT

With the flourishing development of emitting materials, tremendous technological progress has been accomplished. However, they still face great challenges in convenient economical environmental-friendly large-scale commercial production. Herein we designed this hybrid lead/rare earth-free crystals with the large size of 2.7*3.0 cm, an emerging class of high-efficiency luminescent material, (PYE)[Cu_2I_3] (**1**), which emits intense photoluminescence with a high emissive quantum yields of 32.11%. In addition, the results demonstrate that it is possible to obtain regulatable fluorescence intensity by introducing free groups in material design through simple molecular design. The dielectric constant curve remains relatively smooth before melting point. Therefore, it will be an important exploration and effective way for new material design to achieve artificial intelligence luminescent molecules, in this way, the personal customized application can be realized in display and sensing.

1. Introduction

Since ancient times, luminescent materials have existed naturally, thus inspiring people's great wisdom in the application of luminescent materials. For example, the ancient people used fluorescent materials to make various types of luminous ornaments and lighting beads [1–5]. In ancient Greece and Rome, some emperors put it in the palace or in the crown, and some queens and princesses decorated it on jewelry or in the bedroom, using it as a national treasure to promote and praise. Therefore, luminescent materials are important materials that have been pursued for thousands of years [6,7]. With the advancement of science and technology, fluorescent materials no longer rely on natural forms, nor do they fix items that are symbolic of the nobility. It is the basis for high-tech such as electronic information and sensing. Since luminescent materials have been used in a variety of optoelectronic devices and luminescence sensing devices, the discovery of new different types of luminescent materials has been an important task for scientists, for example, the synthesis of various types of luminescent materials, such as red\orange\yellow\green\blue\purple\white and other colors of luminescent materials, as well as various chromaticity of luminescent materials, covering all bands of visible light [1–3,8–15]. However, this is not to say that the task of the scientist is completed. Rather, more scientific approaches are needed to synthesize new materials for different lighting forms and devices. Traditional rare earth luminescence is too expensive, and the natural content of rare earths is limited, which is a world problem. Gold and silver are the same as rare earths.

So in order to find earth-abundant and fabricate easily emitting materials, researchers have paid their attention to highly emissive copper(I) complexes/organic-inorganic hybrid lead(II) halide compound, which have low-cost preparation process and could emit strong light under external stimulus [9–15]. Therefore, the copper and iron with rich content in nature has become a new research focus and breakthrough point. Many researchers have done very interesting work and made significant progress [1–10]. Among the many scientific research work that has been done, the copper and iron has become a research trend. For example, the luminescence of cuprous copper has gradually achieved very rapid progress and improvement. However, the low thermal stability of copper(I) compound, and the high toxicity of organic-inorganic hybrid lead(II) halide compound limit their development in the field of emitting materials [16–20]. In the context of this world voice, our research team has done a series of work. These tasks have taken on the translational work, which provides the important role with multi-elementary. Herein, a stable, highly luminous copper compound is synthesized by in-situ hydrothermal method, which emits strong yellow light under external stimulus and displays high thermal stability (>510 K). These perfect characteristics together with the high emissive quantum yields (32.11%) enable 1 to be

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* Corresponding author.
E-mail address: 247947771@qq.com (Y.-L. Zhang).

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promising candidates for the fabrication of economical environmentally emitting materials [1–5,20–28]. Moreover, the single-crystal X-ray diffraction, thermal analysis, IR spectrum and luminescence measurements have also been performed to characterize the properties of 1, and the crystal structures and optical properties of compound **1** were also been discussed in this paper.

2. Experimental

2.1. Preparation of crystals

All reagents were purchased from commercial sources and used as received. A mixture of $CuCl_2$ (0.11 g, 0.6 mmol), KI (0.117 g, 0.6 mmol), H_2O (8 ml), pyridine (0.5 ml) and EtOH (10 ml) in the molar ratio of 3: 3: 224: 3:120 was stirred for 2 min [1–4]. The resulting solution was transferred into a 25 ml Teflon-lined stainless steel reactor, which was heated at 100 °C for 2 days. After cooling to room temperature, yellow crystals [pye][Cu_3I_4] (**1**) as a single phase were recovered in 54% yield based on Cu^{2+}. And this compound can also be obtained by direct method as shown in scheme of Fig. 1. The molar ratio of the starting materials for both processes was the same except that pyridine was replaced with ethylpyridine hydrochloride. Anal. Calc. for $C_7H_{10}Cu_3I_4N$: C, 10.42; H, 1.24; N, 1.74. Found: C, 10.53; H, 1.23; N, 1.62. IR (KBr, cm^{-1}): 2960w, 1632s, 1566w, 1474 m, 1219 m, 1201w, 1076w, 998w and 654w. The large crystal image with the size of 3.0*2.7 cm was provided in Fig. 1S.

2.2. Single crystal X-ray crystallography

The X-ray single crystal structure for compound **1** was determined with a Rigaku-Qxford Xlab-pro diffractometer at 293 K employing graphite monochromaticed Mo $K\alpha$ radiation ($\lambda = 0.71073$ Å). The data processing including the absorption corrections was processed with Rigaku-Qxford software package. Crystal structure was solved with direct methods and refined on F^2 by means of full-matrix least squares techniques (SHELXL-2014 software package) by using anisotropic displacement parameters. Positions of all H atoms were fixed geometrically and refined via a "riding" model with their U_{iso} values 1.2 times their rider atoms. All the non-H atoms were refined anisotropically by means of all reflections with $I > 2\sigma(I)$. Crystallographic data and structure refinements at 293 K were summarized in Table 1.

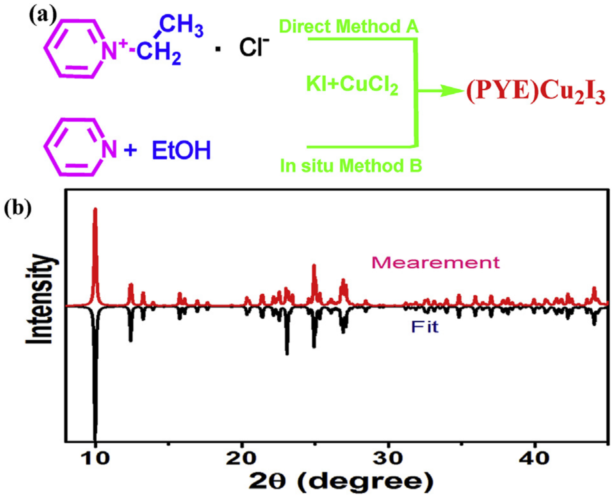

Fig. 1. (a) The schematic presentation of the synthesis of compound **1** with different methods. (b) The comparison of simulated and measured PXRD curves.

Table 1
Crystal data and structure refinements for **1** at 293 K.

Empirical formula	$C_7H_{10}NCu_2I_3$
Formula weight	615.94
Crystal system	monoclinic
Space group	$P2_1/c$
a/Å	8.6567(17)
b/Å	14.184(3)
c/Å	13.727(4)
β/°	125.37(2)
Volume (Å^3)	1374.4(6)
F(000)	1104
Collected reflections	3150
Unique reflections	9210
Absorption correction	Multi-scan
Z, Calculated density	4, 2.977 Mg/m^3
Goodness-of-fit on F^2	1.181
R_1/wR_2 [$I > 2\sigma(I)$]	0.0672/0.1577

2.3. IR spectrum and powder X-ray diffraction measurement

IR spectrum was recorded on a Shimadzu model IR-60 spectrometer at room temperature in the form of KBr diluted pellets. Powder X-ray diffraction data was collected using a Rigaku D/max2200 X-ray diffractometer at 293 K in the 2θ range of 5–40° with a step size of 0.02°. And the powder X-ray diffraction (PXRD) pattern of **1** match pretty well with the pattern simulated from the single crystal structure, which indicates the phase purity (the powder PXRD as shown in Fig. 1b, and the single crystal structure was shown in Fig. 2),

2.4. Luminescence measurements

The measurements of excitation and emission photoluminescence spectra were performed on a Pluorolog 3-TCSPC spectrofluorometer (Horiba Jobin Yvon Inc) equipped with a 450 W xenon lamp as an exciting source, and an emission monochromator, a double-excitation monochromator, and a photomultiplier were used for detection [1–3,25]. The crystal sample was sealed in the quartz cuvette, and then the luminescent quantum yields measurement was performed at room temperature by the integrating sphere (142 mm in diameter).

2.5. Thermogravimetric (TG) analysis

Thermogravimetric (TG) analysis was undertaken on a Dazhan TGA-101 type thermo-analyzer from 303 K to 973 K with a heating rate of 10 K/min under nitrogen atmosphere (Fig. 4a).

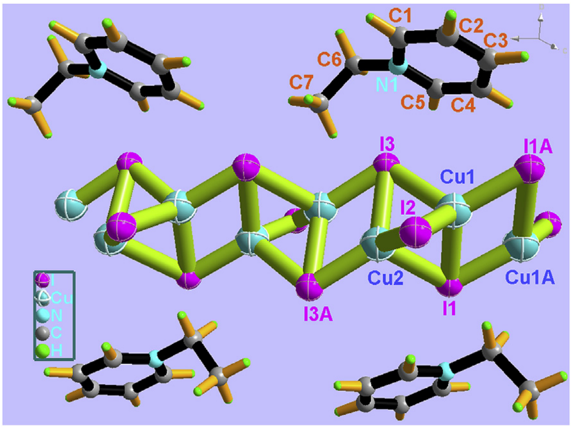

Fig. 2. The packing diagram of **1** exhibited at 293 K.

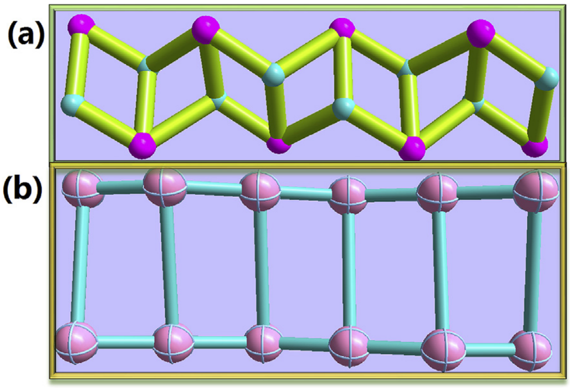

Fig. 3. (a) The zigzag inorganic chain of **1** shown at 293 K, all the H/C/N and part I atoms had been omitted for charity. (b) The simplified ladder-like inorganic chain.

3. Results and discussion

3.1. Thermal properties of 1

In order to investigate the perfect thermal stability of luminescent compound **1**, thermo-gravimetric (TG) was carried out from room temperature to 973 K. As displayed in Fig. 3a, the sample shows a thermal stability up to T = 513 K. What's more, the decomposition of compound **1** mainly was divided into two steps. In the first stage (513–665 K), **1** decomposed into metal(II) iodide. And the second stage (665–973 K) was the procedure in which metal(II) iodide decomposed continuously [1,2]. The TG curve shows a continuous weight loss of 79.4% finally, indicating the removal of cations and iodine. The remaining weight percentage of 20.62% is in agreement with the theoretical value of elemental Cu.

3.2. Crystal structure of 1

In order to explore the emitting mechanism of **1**, a precise analysis to the molecular structure was performed at 293 K. Compound **1** crystallized in the monoclinic space group $P2_1/c$ (*No* 14), and its cell parameters were shown in Table 1. And the asymmetric unit of **1** consists of one independent cation and one $Cu_2I_3^-$ anion unit, as Fig. 2. Single-crystal X-ray structure analysis showed that compound is a 1D inorganic Cu_2I_3 chain, and the cations are anchored to the chain through crystal packing. Therefore, the structure can be divided into two parts: PYE cation units and $(Cu_2I_3)_n$ chains. As shown in Fig. 2, each PYE molecule lies along the inorganic chain. The Cu(1) cation serve as tetrahedral coordination site, but it adopt distortion coordination mode: I–Cu(1)–I angles are 101.200(2), 104.566(6), 118.076(6) and 114.586(6); and Cu–I distances are I1–Cu1 = 2.5466(14), I1–Cu1 = 2.7206(17), I2–Cu1 = 2.6531(16), I3–Cu1 = 2.8338(18), respectively. And for the Cu(2), it also adopt a twisted four-coordinate pattern with I1–Cu2 = 2.940(2), I2–Cu2 = 2.6121(17), I3–Cu2 = 2.5407(15), I3–Cu2 = 2.7836(19), and I–Cu(2)–I angles are 112.099(6),

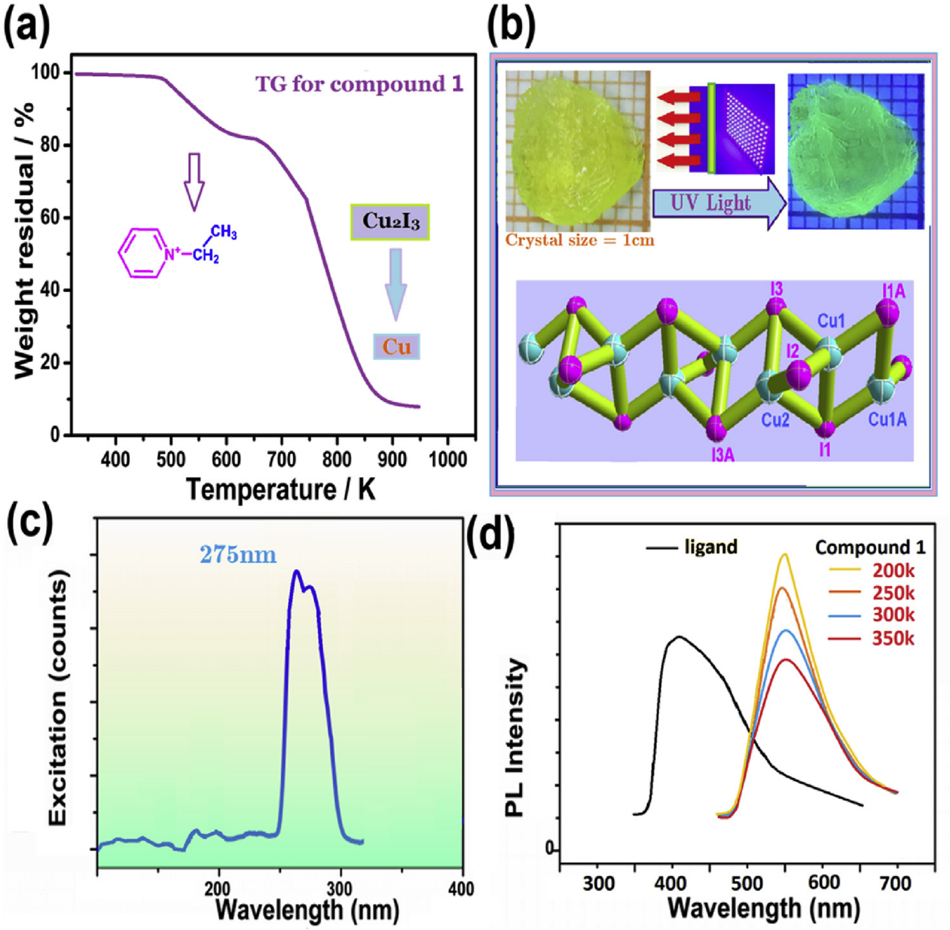

Fig. 4. (a) The TG curve of compound **1** shown the decomposition curve depend on temperature. (b) The yellow block crystal with the size of 1 cm of **1** under sunlight and UV light to display the yellow luminescence, respectively. (c) The excitation spectrum of **1** at room temperature, which has a maximum absorption peak at 275 nm. (d) Emission spectrum of the powder sample at 275 nm excitation, which owns a maximum emission peak at 556 nm. And the yellow-emission intensities were associated with the temperature.

99.774(5), 103.595(5), 96.211(5) and 120.837(6). Obviously, the Cu(2) atom is a more distorted tetrahedral coordination metal with two I3 and one I1/I2 environment. Interestingly, each I1 or I3 anion is coordinated to three copper cations, showing a less common μ_3-I bridging mode, and I2 is a μ_2 mode [1–4,28–31]. Polynuclear complexes, especially Cu(I), are of great interest due to their remarkable photo-physical and catalytic properties in modern material researches. For special d^{10} metal-clusters, the attractive interactions between closed d^{10} shells have received our great attention for the arrangement of metal centers, that is to say, the photo-physical behaviors totally dependent on the number of polynuclear metal atoms. Therefore, the controlled formation of small clusters or low-dimension structures of the metal with defined nuclearities and the study of their structure–function correlations are of great importance in designing functional materials. In this research, one 1D polynuclear Cu(I) luminescent material was designed. The packing of the components is presented in Fig. 2 and Fig. 2S. And the zigzag inorganic chain of **1** was shown in Fig. 3, this simplified inorganic chain just like a ladder.

Dielectric testing indicates that the structure does not exhibit phase transition characteristics. The dielectric constant curve remains relatively smooth before melting point. The *U*eq of the room temperature structure is very reasonable, so there is no structural phase transition in the relatively low temperature state. High temperature dielectric tests have shown that higher temperature still do not trigger significant changes in structure and dielectric. The relevant dielectric test results are presented in the supporting information (see supporting information Fig. 3S).

3.3. *Luminescence properties*

For the potential applications of luminescent materials in modern electronic information industry, many series of fluorescent materials have been designed and synthesized, such as rare earths, d^{10} precious metals, transition metals, and so on. Among them, Cu has been studied due to its luminescent properties and abundant natural resources. Herein, to better characterize the luminescence properties of compound **1**, the excitation and associated emission spectra analysis were depicted as follows. Upon excitation at $\lambda_{ex} = 275$ nm, ligand PYE and its complex show strong emissions at 425 nm and 556 nm, respectively (Fig. 4). Free ligand exhibits strong emission bands in 360–510 nm region upon excitation of $\lambda_{ex} = 275$ nm. This emission may originate from its own conjugated electron transport and $\pi \rightarrow \pi^*$ transition in the packing mode of pyridine rings, which was reported by Li and other researchers [1–10]. The luminescence peaks are completely different for the free ligand/CuI and complex, which told us that there is a weak interaction between the organic and inorganic parts of the complex, and that there are mechanisms for covalent and coordinate bonds to electron transport and fluorescence excitation. Halide-Cu(I) charge transmission here shows the contribution of its electron transport to fluorescence in compound **1**. And the coordination environment and the λ_{max} of the emission of this complex are similar to those of the reported complexes [1–5], which allows enough luminous activators and a higher quantum yield [4,36]. To our knowledge, the quantum yield of 32.11% was fairly high among the reported luminescent molecular-based compounds.

Since the organic group/free cations in the structure have no rigid fixation effect of coordination, its thermal movement with temperature will be significantly higher than that of the coordinated group with rigid fixation [32–36]. That is to say, the organic group acts like a loss of energy as the temperature increases. Therefore, there is a significant temperature effect on the fluorescence intensity. Based on this theoretical basis, we did relative experiments based on the fluorescence intensity with temperature increasing or decreasing to verify the idea. The ideal results we obtained give a solid foundation for the relationship between fluorescence intensity and thermal motion energy loss. Although the organic group does not participate in the coordination, there is a weak van der Waals interaction in the structure, which acts as a medium for energy transfer and plays a leading role in the change of fluorescence intensity. Therefore, the results demonstrate that it is possible to obtain regulatable fluorescence by introducing different free groups in material design, which will enrich the application of fluorescence sensing and intensity probes through simple molecular design. It will be an important exploration and effective way for new material design to achieve artificial intelligence molecules, in this way, the personal customized application can be realized. We will continue to work on this complicated molecular regulation system. The detail information of QE measurement information was submitted in experiment part.

4. Conclusions

The solid sample exhibits yellow emission (556 nm) under UV radiation (275 nm), and the emission intensity shows a quantitative linear relationship with temperature in a wide range from 200 K to 350 K, giving rise to a potential application as a temperature sensor. This work provides a new insight into the understanding of the interactions between Cu_2I_3 and cation units and the photophysical mechanism. Further studies about the influences of different free substituents interactions and photoluminescence properties are ongoing. Dielectric testing indicates that the structure does not exhibit phase transition characteristics. The dielectric constant curve remains relatively smooth before melting point. Such perfect performance of **1** indicates that it would be an excellent candidate for the newly economical environmentally lead/rare earth-free emitting materials and bring light to the development of the results demonstrate that it is possible to obtain regulatable fluorescence by introducing different free groups in material design, which will enrich the application of fluorescence sensing and intensity probes through simple molecular design. It will be an important exploration and effective way for new material design to achieve artificial intelligence molecules.

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Appendix A. Supplementary data

Supplementary data to this article can be found online at https://doi.org/10.1016/j.molstruc.2019.01.084.

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