

Article

2D Layered Uranyl Coordination Framework: Tetracycline Photodegradation and Selective Fe³⁺ Sensing

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Abstract

As a typical representative of antibiotic contaminants, tetracycline (TC) remains persistent in surface water and wastewater. Coordination polymers have been confirmed to represent a highly efficient strategy for pollutant removal. In this study, a novel U(VI)-containing polymer, [UO₂(Htci)]·7.5H₂O, was obtained hydrothermally using uranyl nitrate hexahydrate and tris(2-carboxyethyl) isocyanurate (H₃tci). Structural characterization by single-crystal X-ray diffraction indicated a 2D layered crystalline architecture. The compound is interconnected by 3-connected Htci^{2−} anions to afford a characteristic (6, 3) honeycomb topological network. The ligand displayed a special *cis-cis-trans* conformation, and all carboxylic acid groups were bis-chelating. In addition, the compound was characterized by elemental analysis, FT-IR spectroscopy, powder X-ray diffraction (PXRD), thermal analysis, and photoluminescence spectroscopy. The photodegradation efficiency of TC reached 93.2% after 120 min under irradiation with UV light. At the same time, metal ion sensing of the compound revealed selectivity in recognition of Fe³⁺, with a detection limit of 0.77 mg·L^{−1} being achieved.

Keywords: uranyl coordination polymer; flexible polycarboxylate ligand; photocatalytic degradation; tetracycline; sensing of metal ions

1. Introduction

Coordination polymers (CPs) have attracted wide attention and found widespread applications in numerous industries due to their diverse structural and functional properties [1–4]. An increasing number of coordination polymers (CPs) have been synthesized, with their applications undergoing continuous exploration and development. Although there are now huge crystal databases based on transition metals and lanthanides, research on 5f actinide polymers remains relatively uncommon. Actinides exhibit multiple oxidation states, as their 5f, 6d, 7s, and 7p orbitals readily participate in bonding interactions, thereby giving rise to their exceptionally diverse coordination chemistry. Within the actinide series, uranium stands out as the most intensively studied element, garnering the greatest research interest. Uranium-based coordination polymers not only display unique topological structures and various geometric frameworks [5,6], but also demonstrate significant potential in applications such as photocatalysis [7,8], separation [9], luminescence [10], recognition [11], etc. Uranyl complexes, which are among the most competitive candidates for photocatalytic systems, display intense visible light absorption, outstanding photocatalytic activity, and promising application potential [8,12].



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Uranyl cations (UO_2^{2+}), which serve as the predominant pattern of uranium-based polymers, typically exhibit tetrahedral, pentagonal, and hexagonal bipyramidal coordination geometries. Moreover, the nature of the ligand is critical in dictating the nuclearity and spatial arrangement of the resulting complexes. Uranyl ions show a strong affinity for carboxylic ligands, affording an extensive scope for exploring diverse carboxylic acid derivatives in the construction of uranyl coordination polymers. Polycarboxylates, widely utilized as ligands for uranyl ions, exhibit diverse structures and notable properties, and have demonstrated proven efficacy in constructing uranyl coordination polymers. In particular, among the various polycarboxylic ligands, H_3tci features a flexible tripodal configuration with an isocyanurate ring. The three carboxylate arms ($-\text{CH}_2-\text{CH}_2-\text{COOH}$) of H_3tci may adopt two possible conformations (*cis-cis-cis* or *cis-cis-trans*), regulated by the geometric constraints of metal ions during the self-assembly process. As a highly flexible tripod ligand, H_3tci has gained significant traction in coordination chemistry in recent years [13–17]. In our previous work, H_3tci was employed to construct some uranyl polymers [18–20]; these display diverse structural characteristics and exhibit promising application potentials.

In order to further explore the uranium-based coordination chemistry of H_3tci and develop novel multifunctional polymers, we selected the flexible H_3tci as the organic ligand to construct new uranyl-based coordination polymers and investigate their potential applications. In this study, a two-dimensional uranyl polymer expressed as $[\text{UO}_2(\text{Htci})]\cdot 7.5\text{H}_2\text{O}$ was presented. X-ray crystallography was adopted for crystal structure determination, and the compound was further investigated using IR, PXRD, and thermal analysis, along with an investigation of its luminescence properties. The results demonstrated that the complex featured a honeycomb-like uranyl framework. Furthermore, the photocatalytic performance of the uranyl polymer in degrading TC antibiotics was investigated. The metal ion sensing performance of the uranyl compound was also studied in detail.

2. Materials and Methods

2.1. Synthesis of the Complex

H_3tci (0.0518 g, 0.15 mmol) was added into an 8 mL deionized water solution containing $\text{UO}_2(\text{NO}_3)_2\cdot 6\text{H}_2\text{O}$ (0.0753 g, 0.15 mmol); this was followed by efficient stirring. Afterwards, 200 μL of 10% diluted pyridine was slowly added dropwise to the resulting mixture until the pH was adjusted to 4. The above solution was sealed in a Teflon-lined stainless steel autoclave; this was maintained at 160 $^\circ\text{C}$ for 96 h, and subsequently cooled to 30 $^\circ\text{C}$ at 10 $^\circ\text{C}\cdot\text{h}^{-1}$. Upon completion of cooling, yellow rod-shaped crystals were successfully isolated. Yield: 59% (basis of uranium). Anal. calc for $\text{C}_{12}\text{H}_{28}\text{N}_3\text{O}_{18.5}\text{U}$ (%): C, 19.26; N, 5.61; H, 3.77. Found: C, 19.43; N, 5.77; H, 3.43.

2.2. Photocatalytic Activity Measurements

TC was selected as a model antibiotic pollutant to evaluate the photodegradation activity of the title uranyl polymer toward aqueous solution under UV light (365 nm, 220 V, 30 W) and Xenon lamp (300 W, simulated sunlight). The compound was dispersed in 100 mL of 48 $\text{mg}\cdot\text{L}^{-1}$ TC aqueous solution within a quartz reactor. Dark stirring of the suspension was performed for 30 min to establish adsorption–desorption equilibrium, followed by continuous stirring under UV/Xenon lamp illumination. Periodically, a sample was withdrawn from the reactor and analyzed using a UV-vis spectrophotometer. The corresponding concentrations were calculated based on the standard curve of TC. Then,

the graph of C_t/C_0 changing with time was plotted. The degradation rate, denoted as d , was determined using the formula below.

$$d = \left(1 - \frac{A_t}{A_0}\right) \times 100\% = \left(1 - \frac{C_t}{C_0}\right) \times 100\% \quad (1)$$

d (%): degradation rate; A_t/A_0 : absorbance of the solution at the initial stage or at the t reaction time of the photocatalytic reaction; C_t/C_0 : concentration ($\text{g}\cdot\text{L}^{-1}$) of TC at the initial stage or at the t reaction time. All spectral measurements were repeated three times, and the data employed for graphing represented the average.

2.3. Sensing of Metal Ions

To investigate the metal ion selectivity of the complex, the sample (3 mg) was uniformly dispersed in aqueous metal chloride solution ($\text{M}(\text{Cl})_x \cdot n(\text{H}_2\text{O})$ ($\text{M} = \text{Na}^+, \text{K}^+, \text{Mg}^{2+}, \text{Ca}^{2+}, \text{Mn}^{2+}, \text{Co}^{2+}, \text{Ni}^{2+}, \text{Zn}^{2+}, \text{Cu}^{2+}, \text{Cd}^{2+}, \text{Fe}^{3+}, \text{Cr}^{3+}, \text{Cs}^+, \text{Sr}^{2+}$), 3 mL, $200 \text{ mg}\cdot\text{L}^{-1}$). The suspensions were sonicated for 30 min and then allowed to stand, forming a stable and homogeneous solution. The above solutions were characterized by the fluorescence emission spectra at an excitation of 325 nm. All data for plotting were the average values of three parallel tests.

The Fe^{3+} sensitivity measurements were performed in accordance with the above procedures, excluding variations in ion concentration.

3. Results and Discussion

3.1. Structural Analysis

X-ray single-crystal diffraction analysis showed that the compound crystallizes in the orthorhombic system with the *Pbca* space group. The structure was solved and refined with Olex2 [21], SHELXL-2019 [22] and SHELXT [23], details of the single crystals are listed in Tables S1 and S2 (CCDC number: 2466734).

The asymmetric unit is mainly composed of one Htci^{2-} ligand and a uranyl center. Each uranyl center is coordinated by eight oxygen atoms, displaying an eight-coordinated hexagonal bipyramidal geometric configuration (Figure 1a). With the uranyl $[\text{O1}=\text{U}=\text{O2}]^{2+}$ as the axial direction, the bond lengths are 1.786(10) and 1.8291(11) Å, and the bond angle is $178.8(5)^\circ$. The six equatorial oxygen atoms (O3, O4, O8², O9², O10¹, O11¹), all originating from carboxyl groups, exhibit U-O bond lengths ranging from 2.403(10) to 2.498(11) Å, with an average value of 2.464(10) Å. The valence of U, calculated through the bond lengths around the uranium atom, is 5.79, which accords well with the hexavalent oxidation state of the uranium center.

The ligand Htci^{2-} exhibits a chair-shaped coordination configuration in space (Figure 1b). The carboxyl groups of Htci^{2-} , featuring highly flexible propionyloxy moieties, adopt a *cis-cis-trans* coordination configuration. The angles between $-\text{CH}_2\text{COO}^-$ and the triazine ring plane are 109.0, 110.2, and 112.2° . However, the reported uranyl complexes based on H_3tci represent *cis-cis-cis* [18,19] and *cis-cis-trans* [19,20] configurations. It has been demonstrated that the carboxylate groups in H_3tci are distorted, and this makes H_3tci a feasible ligand for forming uranyl coordination polymers with diverse spatial structures and topologies. Three carboxyl groups of H_3tci are fully deprotonated. Three propionic acid groups of H_3tci keep turning and extending in space, forming an ABAB-packing two-dimensional spatial wavy structure along the *bc* plane (Figure 1c). Each tripodal Htci^{2-} ligand binds to three UO_2^{2+} metal centers, and each of the metal centers is bonded to three Htci^{2-} ligands. These are interconnected and expanded along the *ac* plane, forming a honeycomb-typed structure. Two layers along the *ac* plane do not completely overlap with each other, but form a two-dimensional spatial layered structure with staggered overlap.

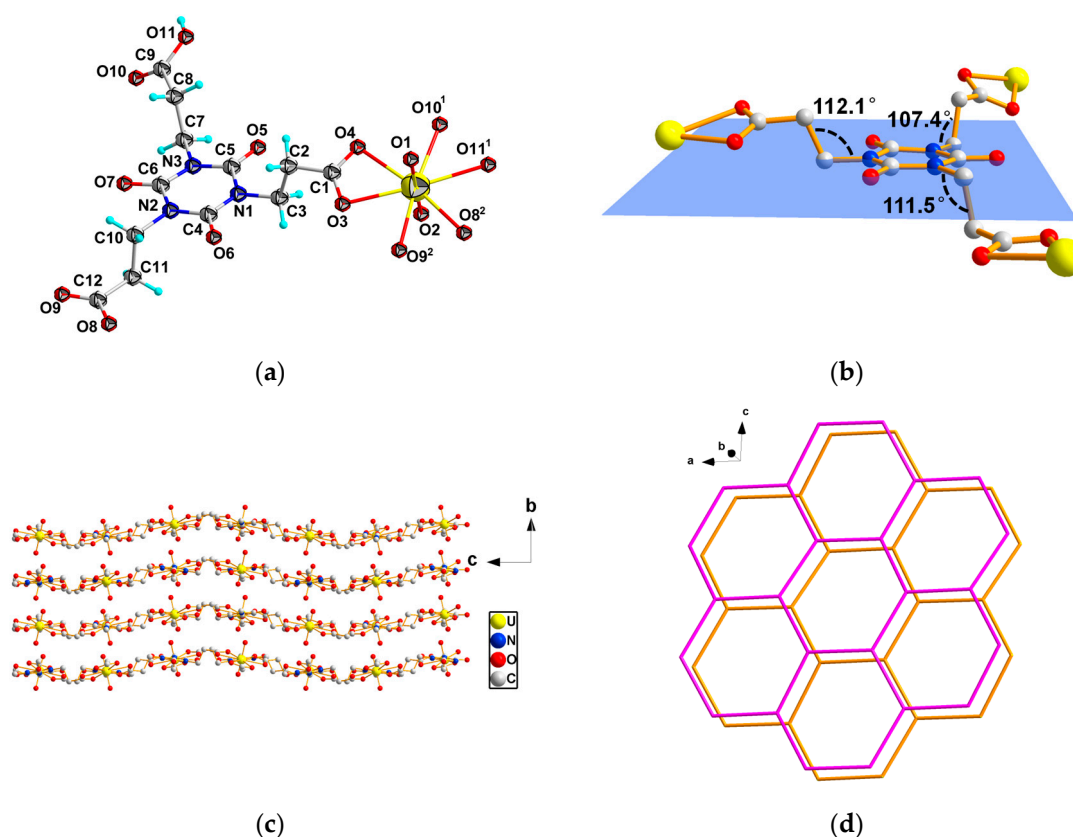


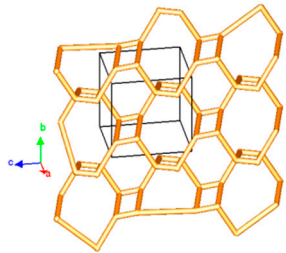
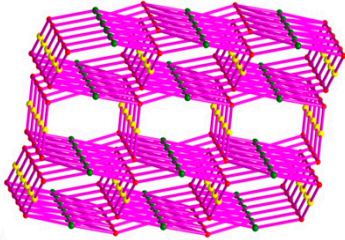
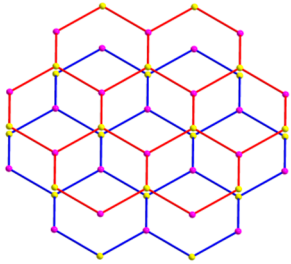
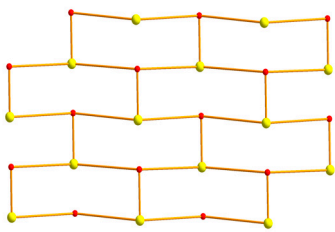
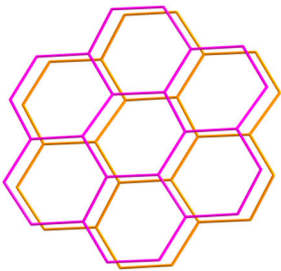
Figure 1. (a) Asymmetric unit with symmetry codes: ¹ 1 - x, -1/2 + y, +3/2 - z; ² 1/2 - x, -1/2 + y, z; (b) Arms of the ligand twisted in the space showed different bond angles; (c) 2D layered structure and adopted ABAB packing mode; (d) Layered honeycomb-like networks of the complex.

To further analyze the topology of the polymer, the Htci²⁻ ligand is simplified as a 3-connected Y-type node to bridge three UO₂²⁺ metal centers. Similarly, each UO₂²⁺ serves as a three-connected node linking three corresponding ligands. The skeleton of the complex comprises a (6, 3) honeycomb-like network and spreads throughout the *ac* plane (Figure 1d). The honeycomb-like polymers of uranyl are not unique because of the non-extensible UO₂²⁺. Some tripodal ligands have a good tendency to construct honeycomb-like structures [18,24–27]. Table 1 lists the structural formulas for all reported examples in uranyl complexes with Htci²⁻, as well as illustrations of the spatial configurations. The complexes in Table 1 differ in terms of their counterions and the pH conditions during the synthesis process, both of which obviously play structure-directing roles. The formation of such structural topological variations may not be regarded as surprising, given the flexible ligand and the capping effect of UO₂²⁺.

3.2. Structural Characterization

FT-IR spectra of the compound and H₃tci are displayed in Figure 2a. Compared with the uncoordinated ligand, the uranyl complex exhibits characteristic absorption maxima at approximately 1039 and 926 cm⁻¹, which correspond to the *v*_{sym} (U=O) and *v*_{asym} (U=O) vibrations, respectively. The disappearance of a band at 1695 cm⁻¹, typical of the free carboxylic acid moiety in H₃tci, verifies the full deprotonation of the carboxylate groups and their coordination to the uranyl centers. The strong absorption bands at 1549, 1462, and 1427 cm⁻¹ in the complex are assigned to the *v*_{asym} and *v*_{sym} of the coordinated carboxylate groups. Additionally, the bands at 768, 683, and 518 cm⁻¹ are ascribed to the skeletal vibrations of the nitrogen-containing triazine rings. These spectral characteristics are consistent with the coordination modes determined by X-ray diffraction.

Table 1. Overview of spatial configurations in uranyl complexes with H₃tci.

Compound	Configurations	Ref.
$\text{UO}_2(\text{tci})(\text{C}_3\text{H}_5\text{N}_2) \cdot \text{H}_2\text{O}$	 2D irregular layered structure	[18]
$(\text{UO}_2)_4(\text{tci})_2(\text{OH})_4[(\text{UO}_2)_2(\text{H}_2\text{O})_4]_{1/2}$	 3D extended network	[19]
$[(\text{UO}_2)_2(\text{tci})_2]_{1/2} \cdot [\text{Mn}_2\text{O}(\text{H}_2\text{O})_8]_{1/2} \cdot 2\text{H}_2\text{O}$	 2D crossed honeycomb-like network	[20]
$[\text{UO}_2(\text{tci})]^- \cdot (\text{C}_{10}\text{H}_9\text{N}_2)^+ \cdot \text{H}_2\text{O}$	 2D wave-like layered structure	[24]
$[\text{UO}_2(\text{Htci})] \cdot 7.5\text{H}_2\text{O}$	 2D overlapping honeycomb-like network	This work

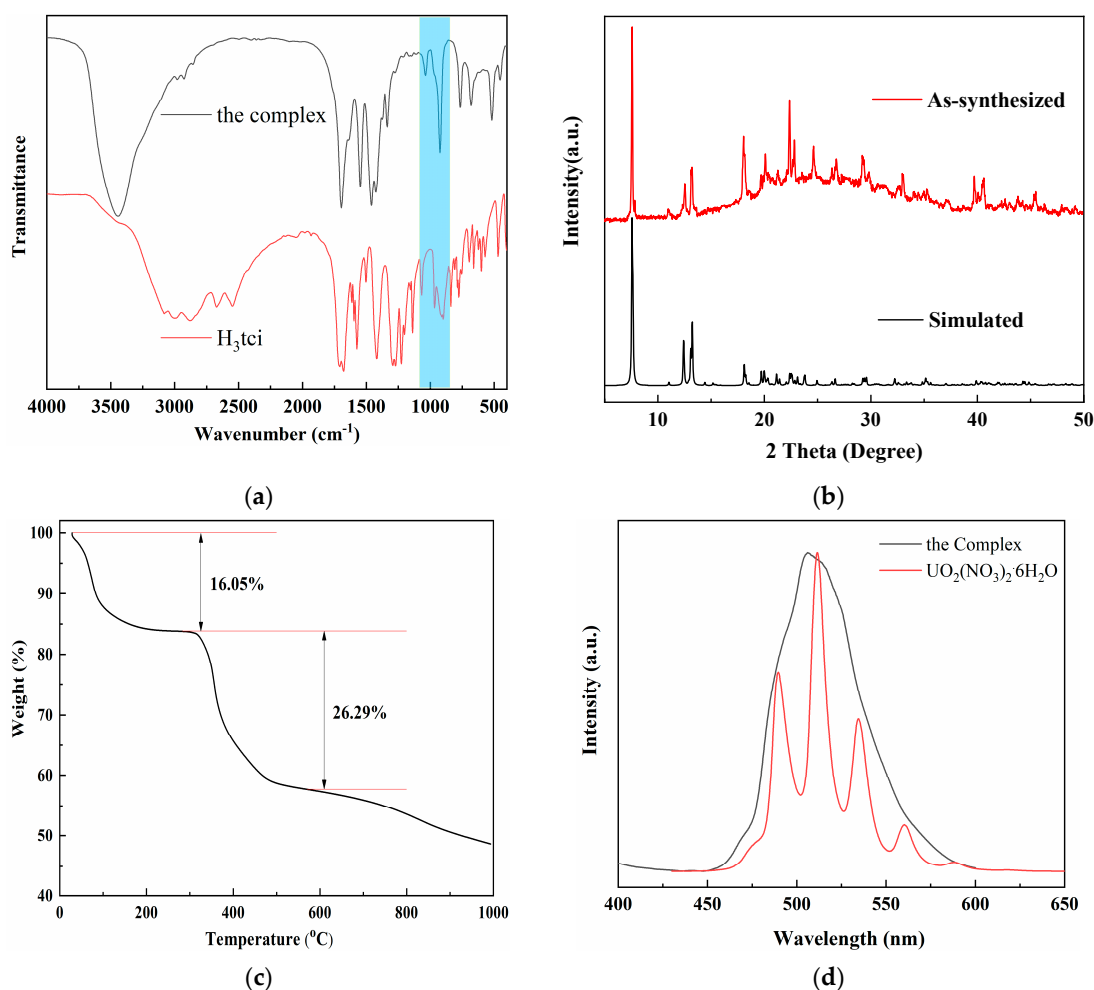


Figure 2. (a) FT-IR spectra of the complex and H₃tci. (b) Simulated PXRD patterns from single-crystal data and experimental patterns of the as-synthesized compound. (c) TG curves of the complex. (d) Emission spectra of the complex and UO₂(NO₃)₂·6H₂O.

The PXRD pattern of the polymer was collected under room temperature (Figure 2b). The synthesized compound shows a close match with the simulated crystal diffraction pattern. The minor discrepancies in peak intensities are likely due to variations in crystallinity introduced during sample preparation. Thus, the sample is suitable for the following investigations.

To assess the thermal stability of the uranyl polymer, thermo-gravimetric measurement was performed under a N₂ atmosphere from 25 °C to 1000 °C at 10 K·min⁻¹ (Figure 2c). The TG curve indicates that the uranyl compound experiences two distinct weight-loss stages. The first step in the range of ambient temperature to 305 °C is ascribed to the loss of free water molecules (obsd: 16.05%; calcd: 18.05%). The second rapid weight-loss step, corresponding to the collapse of the network, takes place between 320 and 582 °C. The second stage is poorly resolved, owing to the partial decomposition of the polymeric structure. The remaining 48.50% might be U₃O₈ (calcd: 1/3U₃O₈, 37.51%).

The solid-state fluorescence emission measurement of the polymer was carried out at ambient temperature under 330 nm excitation (Figure 2d). The emission spectrum measured in the range of 400–600 nm was compared with that of uranyl nitrate hexahydrate. In contrast to the characteristic emission peaks of uranyl nitrate (476, 489, 511, 534, 560, 588 nm), the polymer exhibits less resolved emission features. The spectrum of the polymer converges as a wide and unsmooth band spanning 460–580 nm, with a central wavelength at 506 nm indicating a slight blue shift of approximately ~5 nm relative to uranyl nitrate

hexahydrate. It is not uncommon for uranyl polymers to have such broad fluorescence emission bands because of the diversity of uranyl structures [28–30].

3.3. Photocatalytic Performance

The presence of UO_2^{2+} in the polymer led to the attractive photocatalytic activities. To evaluate the catalytic degradation efficiency of the target polymer, TC was chosen as a representative contaminant under both UV-light and xenon-lamp irradiation.

The degradation process of TC in aqueous solution was monitored in the presence of the target polymer. After being irradiated by UV lamp for 120 min, the photocatalytic degradation rates of TC containing the different concentrations of uranyl polymer (2, 4, 6, 8, and $10\text{ mg}\cdot\text{L}^{-1}$) were 39.8, 60.7, 64.9, 76.8, and 93.2%, respectively (Figure 3a). Taking into account both the photocatalytic degradation efficiency and the catalyst concentration, the optimal polymer dosage was determined to be $10\text{ mg}\cdot\text{L}^{-1}$ for the purposes of further experiments. Under xenon-lamp irradiation, the photocatalytic degradation rate of the polymer for TC was 52.1% at 120 min (2.9% no catalyst under xenon lamp). Meanwhile, under irradiation with UV light, the degradation rate was 93.2% at 120 min (11.7% no catalyst under UV light) (Figure 3b). The decreasing UV-Vis absorption spectra of the TC under Xenon lamp and UV light are displayed in Figure 3c,d. These results clearly demonstrate that the uranyl-based polymers exert a considerable effect on the photodegradation of TC [25,31].

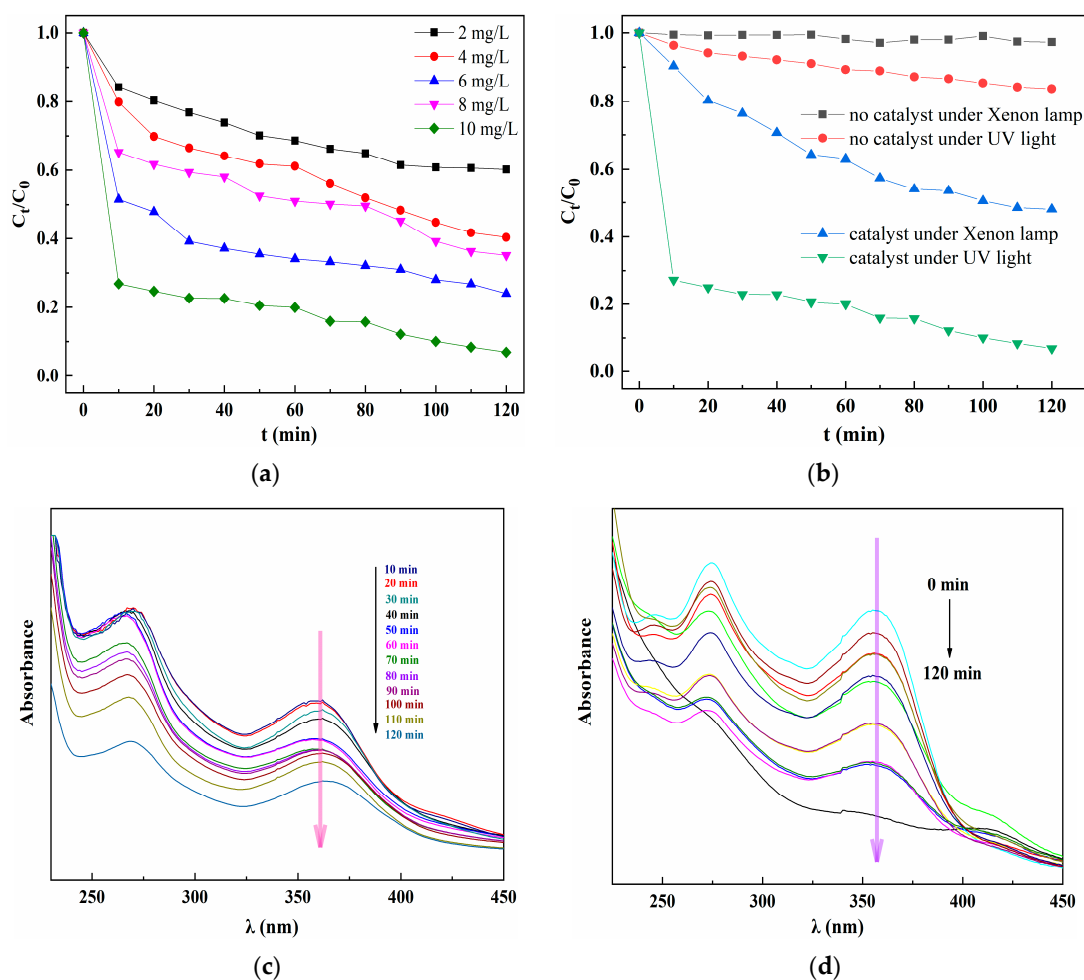


Figure 3. (a) Photocatalytic degradation of TC with varying concentrations of the uranyl polymer. (b) Photocatalytic degradation of TC under xenon-lamp and UV-light irradiation. (c,d) Time-dependent UV-Vis absorption spectra of TC during degradation under a xenon lamp (c) and UV light (d).

3.4. Sensing Property for Fe^{3+}

Fluorescence spectra of the complex in the presence of diverse metal ions exhibited similar profiles to the blank sample. No obvious shifts in peak positions or variations in peak shapes were observed; however distinct changes in fluorescence intensity did occur. Fluorescence intensity increased markedly in Cd^{2+} , remained nearly unchanged in K^+ , Zn^{2+} and Sr^{2+} , decreased slightly in Na^+ , Mg^{2+} , Mn^{2+} , Ni^{2+} and Cr^{3+} , dropped obviously in Ca^{2+} , Co^{2+} , Cu^{2+} and Cs^+ , and was almost fully quenched in Fe^{3+} (Figure 4a,b). The results show that the complex exhibits a sharp quenching of fluorescence emission intensity upon the addition of Fe^{3+} , indicating high selectivity for Fe^{3+} among the various metal cations.

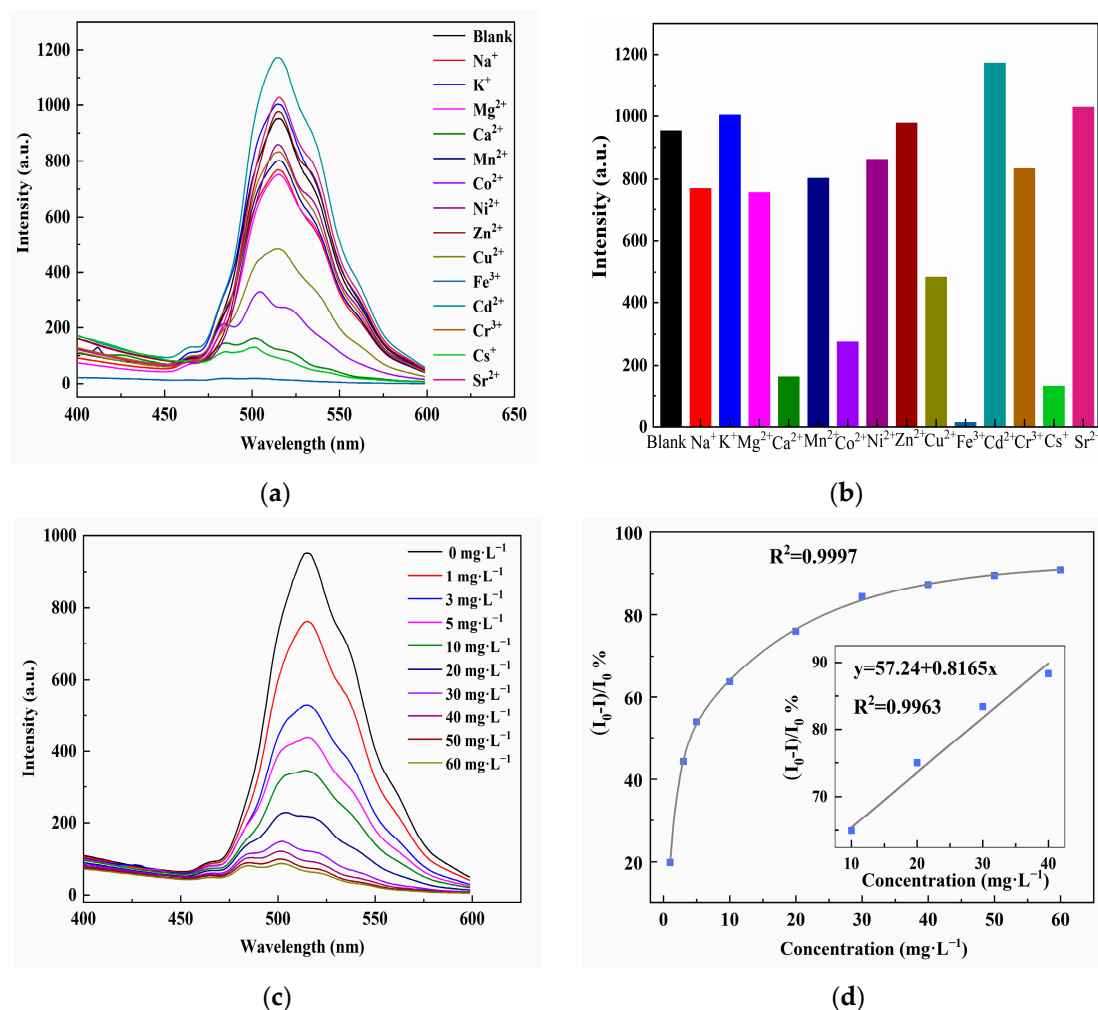


Figure 4. (a,b) Emission spectra and intensities of the complex dispersed into different metal salt solutions. (c) Emission spectra of the complex at various Fe^{3+} solutions (0–60 $\text{mg}\cdot\text{L}^{-1}$). (d) Simulated correlation between $(I_0 - I)/I_0\%$ and Fe^{3+} concentration; the inset displays the linear fitting range from 10 to 40 $\text{mg}\cdot\text{L}^{-1}$.

Based on the selectivity of the complex for sensing Fe^{3+} , further studies of its sensitivity under different Fe^{3+} concentrations were conducted. The quenching constant (K_{sv}) was obtained using the Stern–Volmer equation, $I_0/I - 1 = K_{sv}[C]$. The calculated K_{sv} value for Fe^{3+} reached 3.35×10^5 , further demonstrating the high sensitivity and selectivity of the compound toward Fe^{3+} . It is interesting to note that the increase in Fe^{3+} concentration from 0 to 60 $\text{mg}\cdot\text{L}^{-1}$ leads to a rapid decline in luminescence intensity (Figure 4c). A correlation analysis between the luminescence quenching ratio $[(I_0 - I)/I_0\%]$ and the Fe^{3+} content was performed. The Langmuir fitting ($R^2 = 0.9997$) confirmed the effective response of

the sensor. Notably, a well-fitted linear relationship ($y = 57.24 + 0.8165x$, $R^2 = 0.9963$) was observed in the concentration range of 10–40 $\text{mg}\cdot\text{L}^{-1}$ (Figure 4d). This sensing method exhibits high sensitivity toward Fe^{3+} over a detection range of 0–60 $\text{mg}\cdot\text{L}^{-1}$, with the limit detection calculated to be 0.77 $\text{mg}\cdot\text{L}^{-1}$, according to the $3\sigma/k$ criterion.

Fluorescence quenching of complex materials by metal ions occurs mainly via three mechanisms: structure disruption, central cation exchange, and competitive light absorption between adsorbed metal ions and the complex. SEM and EDS results confirm that iron species are loaded on the compound (Figure 5a). EDS mapping shows the elemental distribution on the surface of the complex (Figure 5b,c), with the atomic% of Fe being 6.84% (Figure 5d). The characteristic absorption peaks in the FTIR spectra demonstrate that the basic structure of the complex remains unchanged after catalysis (Figure S2). Consistently, the PXRD results further confirm that no obvious structural variation occurs in the complex after the catalytic reaction (Figure S3). Accordingly, the fluorescence quenching is attributed to competitive light absorption and energy transfer between Fe^{3+} and uranyl complex [20]. The excitation spectrum of the uranyl complex partially overlaps with the absorption spectrum of Fe^{3+} . The dominant portion of excitation energy is taken up by Fe^{3+} loaded on the material surface. This effect reduces the absorption intensity of the complex, resulting in reduced luminescence or even total quenching.

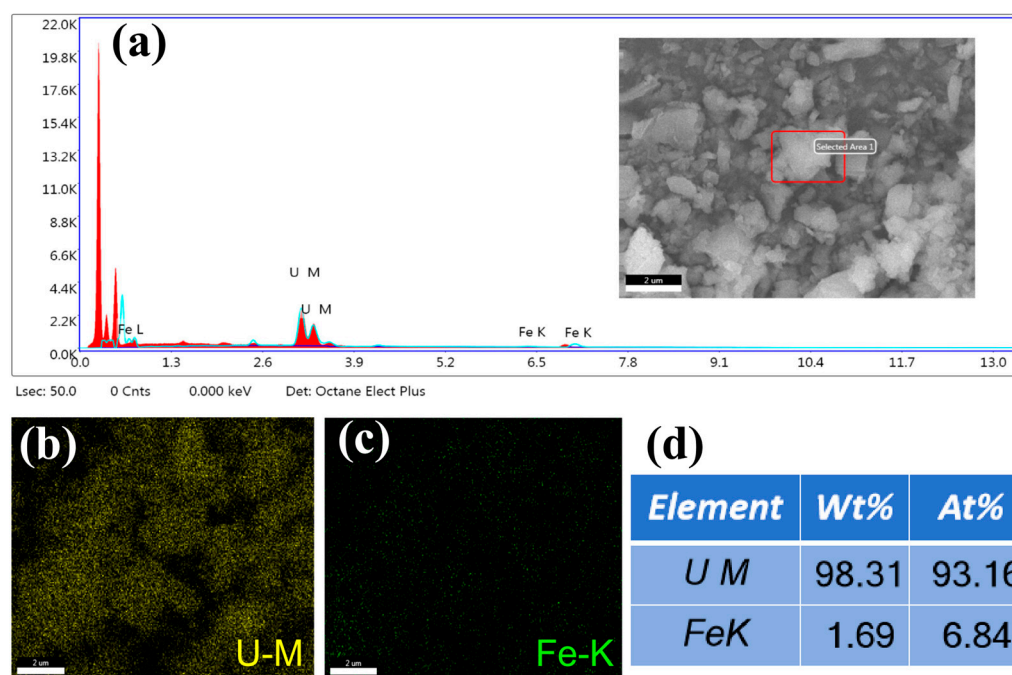


Figure 5. (a) EDS analysis of the Fe loaded on the compound; inset is the SEM photograph of the compound. (b,c) EDS mapping of U and Fe elements showed the elemental distribution on the surface. (d) Results of EDS analysis of the compound for U and Fe.

4. Conclusions

A high-purity uranyl polymer constructed from H_3tci was successfully prepared via a hydrothermal route. Structural characterization indicates that the polymer features a honeycomb-like 2D layered structure, with the ligand adopting a *cis-cis-trans* conformation. The luminescent property suggests that the polymer can be conducted as a potentially fluorescent material. Notably, the polymer exhibits remarkable photocatalytic activity toward TC degradation under UV irradiation. The photodegradation efficiency of TC solution reached about 93.2% in 120 min, which provides the theoretical and experimental basis for the application of uranyl polymers. Meanwhile, the metal ion sensing of the

compound revealed a selective recognition of Fe^{3+} , with a detection limit of $0.77 \text{ mg} \cdot \text{L}^{-1}$. These results establish the practical foundation for utilizing the uranyl polymer as an efficient photocatalyst in treatment of antibiotic-contaminated wastewater.

Supplementary Materials: The following supporting information can be downloaded at: <https://www.mdpi.com/article/10.3390/cryst16070443/s1>, Table S1: Crystal data and structure parameters of the complex; Table S2: Selected bond lengths (Å) and angles (deg) of the complex; Figure S1: The solid-state UV-vis spectra of the uranyl complex; Figure S2: FT-IR spectra comparison of the complex before and after photocatalysis; Figure S3: PXRD patterns comparison of the complex before and after photocatalysis.

Author Contributions: Conceptualization, L.-L.L.; methodology, L.-L.L.; formal analysis, Z.-Y.L., T.-T.L. and Y.-Z.Z.; investigation, Z.-Y.L., T.-T.L. and Y.-Z.Z.; writing—original draft preparation, L.-L.L.; writing—review and editing, L.-L.L.; project administration, L.-L.L. and J.-S.Z. All authors have read and agreed to the published version of the manuscript.

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Data Availability Statement: Crystallographic data for the uranyl complex (CCDC number: 2466734) can be obtained free of charge via http://www.ccdc.cam.ac.uk//data_request/cif (accessed on 3 May 2026).

Conflicts of Interest: The authors declare no conflicts of interest.

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