Synthesis, structure, fluorescence, electrochemical, and antioxidant studies of a new silver(I) complex with 2,6-bis(N-methylbenzimidazol-2-yl)pyridine

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Abstract
A new silver(I) complex of the type [Ag₂(mbBP)₂](pic)₂·DMF·CH₃OH [mbBP = 2,6-bis(N-methylbenzimidazol-2-yl)pyridine] is synthesized and analyzed using a range of spectroscopic and crystallographic techniques. This compound contains cationic Ag₂(mbBP)₂ dimers, in which the crossed eight-shaped structure is formed by two mbBP ligands bridging two silver(I) centers and Ag–Ag bonds. Two-dimensional supramolecular networks of the silver(I) complex are connected by π···π interactions and hydrogen bonds. Hirshfeld analyses are performed to support the aforementioned intermolecular interactions. The effect of π···π stacking interactions on the fluorescence properties of the complex is investigated. Cyclic voltammetry indicates two irreversible redox processes. In addition, an antioxidant assay in vitro demonstrates that the silver(I) complex displays excellent scavenging activity toward hydroxyl radicals.

Keywords
antioxidant, crystal structure, fluorescence, silver(I) complex, supramolecular interactions

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Introduction
In recent years, considerable research efforts have been devoted to the study of silver(I) complexes with nitrogen heterocyclic ligands due to their fascinating geometric structures, interesting properties, and medicinal applications.1–6 Different from other transition-metal ions, Ag(I) has a tendency to display a highly versatile and irregular coordination number (2–8) because of its intrinsic electronic configuration, leading to the formation of various types of coordination architectures such as linear, trigonal, tetrahedral, square planar, trigonal pyramidal, and octahedral.7–9 A wide variety of nitrogen heterocyclic ligands have been used to synthesize silver(I) complexes, including benzimidazole, imidazole, pyridine, pyrrole, benzoxazole, pyrazine, quinolone, and their derivatives.10–12

In the literature and according to our previous work, it is found that V-shaped bis(benzimidazole) ligands are capable of chelating silver ions to form stable complexes.13–16 In addition, the presence of amino groups on benzimidazole can lead to the formation of hydrogen bonds with either nitrogen atoms or oxygen atoms or solvent molecules as well as anions.15 Moreover, benzimidazole contains an aromatic ring that plays different roles in the structure and packing of the new compounds, for example, π···π stacking interactions.18,19 However, there are few reports on the relationship between supramolecular effects and properties, especially in terms of fluorescence properties.

Herein, we have selected a V-shaped ligand, 2,6-bis(N-methylbenzimidazol-2-yl)pyridine, which can provide three nitrogen atoms as coordination sites for the synthesis of a binuclear Ag(I) complex. The complex was structurally characterized by elemental analysis, infrared (IR), ultraviolet visible (UV-Vis) spectroscopy, and single-crystal X-ray diffraction. The fluorescence and antioxidant activities of the Ag(I) complex have been investigated. We focused on the correlation between the supramolecular π···π conjugation and the fluorescence quenching of the Ag(I) complex.

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Results and discussion

The complex was obtained by the reaction of Ag(pic) (pic = picric acid) with mbbp (mbbp = 2,6-bis(N-methylbenzimidazol-2-yl)pyridine) in methanol and dimethylformamide (DMF). They are soluble in polar aprotic solvents, such as DMF and dimethyl sulfoxide (DMSO), slightly soluble in ethanol and methanol, and insoluble in Et2O and petroleum ether. Strong and broad absorption bands were observed around 1483 and 1278 cm\(^{-1}\) in the IR spectrum of the free ligand, indicating the presence of \(\nu(C=N)\) and \(\nu(C–N)\) due to the benzimidazole, which become medium bands around 1473 and 1263 cm\(^{-1}\) for the complex.\(^{20}\) The shifts of the characteristic absorption bands imply that the mbbp ligands are coordinated to the cation center.\(^{21–24}\) The electronic spectra of free mbbp and the complex were recorded at 298 K in DMF. The mbbp ligand shows one strong absorption band at 324 nm, which is assigned to \(\pi–\pi^*\) transitions.\(^{25,26}\) The complex exhibits two strong absorption bands (325 and 380 nm), and the band due to free mbbp is slightly red-shifted in the complex indicating that the mbbp ligand is coordinated to the Ag(I) ions.\(^{27}\) The new absorption of the complex at 380 nm is attributed to the \(\pi–\pi^*\) (picrate) transitions.\(^{28,29}\)

X-ray structure determination

The Ag(I) complex crystallizes in the triclinic space group \(P\bar{1}\). Single-crystal X-ray analysis revealed that the asymmetric unit consists of an Ag\(_2\)(mbbp) dimer, two picate anions, and molecule each of one methanol and DMF. The Ag\(_2\)(mbbp) dimer shows a crossed eight-shaped structure, which is formed by two mbbp ligands bridging two silver(I) centers and Ag–Ag bonds (Figure 1(a) and (b)). As shown in Figure 1(c), each dimer contains two silver atoms with an Ag–Ag distance of 2.9915(3) Å, which is within the reported Ag–Ag bond range of 2.85–3.29 Å.\(^{30}\) The two Ag atoms are both coordinated by three nitrogen atoms from two mbbps and the coordination geometry may best be described as a distorted planar triangle. The Ag–N distances are in the range of 2.126(2)–2.752(2) Å and the values are in accordance with those previously reported for other silver complexes with aromatic N-heterocycles.\(^{31}\) Each mbbp coordinates to two Ag(I) atoms in the \(\eta^1:\eta^1:\eta^1:\mu_2\) bridging mode (Figure 1(d)), which is quite different from those reported in the literature. The binding mode of mbbp usually adopts tridentate chelation coordination with other transition-metal centers.\(^{32,33}\) The picate anions do not participate in the coordination and only act as counter anions for charge equilibrium.

As shown in Figure 2, the two Ag\(_2\)(mbbp) dimers sandwich the two picrates through \(\pi–\pi\) conjugation (centroid-to-centroid distances: 3.394 and 3.365 Å) to form a supramolecular one-dimensional (1D) polymer. The adjacent 1D chains are linked into a two-dimensional (2D) network structure by C–H\(\cdot\cdot\cdot\)O hydrogen bonding.

Hirshfeld surface analysis

The nature of the \(\pi–\pi\) and C–H–O interactions in the complex are analyzed from the Hirshfeld surfaces using the Crystal Explorer 17.5 program.\(^{34–36}\) As shown in Figure 3(a), the red areas are attributed to \(\pi–\pi\) interactions. The larger red spots in Figure 3(b) are attributable to the presence of C–H–O hydrogen bonds. The \(\pi–\pi\) and C–H–O contacts contributed 4.4% and 31.1% to the sum of all the intermolecular interactions, respectively, appearing as spikes in the 2D fingerprint plot (Figure 3(c) and (d)), indicating that these interactions are significantly strong.\(^{37}\) These results provide further evidence of the existence of \(\pi–\pi\) stacking interactions and hydrogen bonds in the complex, which is consistent with X-ray crystallography results.
Figure 2. 2D network formed via $\pi \cdots \pi$ and C–H–O interactions in the complex (different interactions are distinguished by different colors).

Figure 3. Hirshfeld surfaces mapped with $d_{norm}$ of the complex ((a) $\pi \cdots \pi$ interactions and (b) C–H–O interactions). 2D fingerprint plot for the complex ((c) $\pi \cdots \pi$ interaction and (d) C–H–O interactions).
Fluorescent properties

The fluorescent spectra of the mbbp and the Ag(I) complex were measured in the solid state at room temperature, as shown in Figure 4. The free ligand mbbp shows purple fluorescence with a maximum wavelength at 477 nm when excited at 350 nm, which is assigned to the $\pi-\pi^*$ transition. The fluorescence of the Ag(I) complex showed full quenching. This may be attributed to the $\pi-\pi$ stacking interactions between the picrate and benzimidazoles in the complex, which was confirmed from the crystal structure and Hirshfeld surface analysis. Our previous research indicated that the presence of $\pi-\pi$ conjugation between benzimidazoles in complexes can cause partial fluorescence quenching. In this work, this is the first time that we have found that the $\pi-\pi$ conjugation leads to complete quenching of fluorescence, which may be related to picric acid that is often used as a fluorescence quenching reagent. The observations provide evidence for the fluorescence quenching mechanism of picrate.

Antioxidant activities

Figure 5 shows the plots of the hydroxyl radical scavenging effect (%) for the mbbp ligand and the complex. The 50% inhibitory concentration ($IC_{50}$) values of mbbp and the complex are $1.4 \times 10^{-5}$ and $5.1 \times 10^{-5} \text{M}$. We compared their abilities to scavenge hydroxyl radicals with the well-known natural antioxidants mannitol, vitamin C, and other reported Ag complexes, using the same method. The $IC_{50}$ values of mannitol, vitamin C, $\text{[Ag(BBO)}_{1/2}(o$-coumarate)]$ \text{DMF}_{\infty}$, $\text{[Ag(bbtt)(crotonate)]CH}_2\text{CH}_2\text{OH}$, $\text{[Ag(bmbt)(p-coumarate)]2CH}_3\text{OH}$ are $9.6 \times 10^{-3}$, $8.7 \times 10^{-3}$, $1.46 \times 10^{-3}$, $4.3 \times 10^{-3}$, and $0.38 \times 10^{-5} \text{M}$, respectively. This indicates that mbbp and the complex exhibit good hydroxyl radical scavenging activity.

Electrochemical studies

The electrochemical properties of the complex were studied by cyclic voltammetry (CV) in DMF. The data are collected in Table 1 and the voltammogram is shown in Figure 6. The Ag(I) complex exhibits two electric pairs, which are attributed to the $\text{Ag}_2$(mbbp)$^{2+}$/Ag(mbbp)$^+$ and Ag(mbbp)$^+$/Ag couples. The separation between the cathodic and anodic peak potentials $\Delta E$ ($\Delta E_{pa} - E_{pc}$) and the current $I$ ($|i_{pa}/i_{pc}|$) indicate that both electric pairs are irreversible redox processes and the first electric pair is less reversible. In addition, the neutral-free mbbp ligand is not electrochemically active over the range of $-1.5$ to $+1.5 \text{V}$.

Conclusion

In summary, a new silver(I) complex has been synthesized and characterized. Structural analysis indicates that the Ag(I) complex is a binuclear structure, which is formed by two mbbp ligands bridging two silver(I) centers. The structure is extended into 2D supramolecular

![Figure 4. The solid-state fluorescent emission spectra of mbbp and the complex.](image)

![Figure 5. Plots of the antioxidation properties of (a) mbbp and (b) the complex in hydroxyl radical scavenging (%).](image)

![Table 1. Electrochemical data of the Ag(I) complex.](table)

| Compound  | Electric pair                  | $E_{pc}$ (V) | $E_{pa}$ (V) | $\Delta E$ (V) | $E_{1/2}$ (V) | $i_{pa}$ ($\mu A$) | $i_{pc}$ ($\mu A$) | $I$  |
|-----------|-------------------------------|--------------|--------------|----------------|--------------|------------------|------------------|-----|
| Ag(I)     | $\text{Ag}_2$(mbbp)$^{2+}$/Ag(mbbp)$^+$ | 0.412        | 0.591        | 0.179          | 0.502        | 45.02            | -9.82            | 4.58 |
| Complex   | $\text{Ag}(\text{mbbp})^+/\text{Ag}$ | 0.084        | 0.415        | 0.331          | 0.248        | 20.53            | -29.98           | 0.69 |

$\Delta E = E_{pa} - E_{pc}; E_{1/2} = (E_{pa} + E_{pc})/2; I = |i_{pa}/i_{pc}|$. 

Figure 4. The solid-state fluorescent emission spectra of mbbp and the complex.

Figure 5. Plots of the antioxidation properties of (a) mbbp and (b) the complex in hydroxyl radical scavenging (%).

Table 1. Electrochemical data of the Ag(I) complex.
frameworks through \( \pi \cdots \pi \) stacking interactions and hydrogen bonds. Fluorescent experiments indicate that the Ag(I) complex undergoes complete fluorescence quenching in the solid state, which is due to the strong \( \pi \cdots \pi \) stacking interactions between picrate and benzimidazole rings. Moreover, the Ag(I) complex exhibited potential antioxidant activity against hydroxyl radicals’ in vitro studies. Electrochemical studies show two irreversible redox behaviors for the Ag(I) complex. These findings indicate that the silver complex \([\text{Ag}_2(\text{mbbp})_2]\) \((\text{pic})_2\)\(\text{DMF-CH}_3\text{OH}\) has potential practical applications in fluorescence and in antioxidant materials.

**Experimental**

**Materials and general methods**

All chemicals and solvents were reagent grade and were used without further purification. C, H, N, and O elemental analyses were performed using a Carlo Erba 1106 elemental analyzer. The IR spectra were recorded in the 4000–400 cm\(^{-1}\) region with a Nicolet FT-VERTEX 70 spectrometer using KBr pellets. Electronic spectra were obtained using a Lab-Tech UV BlueStar spectrophotometer. The absorbance was measured with a Spectrula 722sp spectrophotometer at room temperature. Fluorescence measurements were performed on an F-7000 FL Spectrophotometer. The hydroxyl radical studies were performed in water-bath with a 722sp spectrophotometer. Further details on the hydroxyl radical scavenging are provided in the Supplemental material. Electrochemical measurements were performed on a LK2005A electrochemical analyzer under nitrogen at 283 K. A glassy carbon working electrode, a platinum-wire auxiliary electrode, and an Ag/AgCl reference electrode ([Cl\(^-\)] = 1.0 mol/L) were used in the three-electrode measurements. The electroactive component was at \(1.0 \times 10^{-3}\) mol dm\(^{-3}\) concentration with tetrabutylammonium perchlorate (TBAP) (0.1 mol dm\(^{-3}\)) used as the supporting electrolyte in DMF.

**Synthesis of the ligand and [Ag\(_2\)(mbbp)]\((\text{pic})_2\)\(\text{DMF-CH}_3\text{OH}\)**

The mbbp ligand was synthesized according to the reported method. The Ag complex was synthesized by the

| Complex | \([\text{Ag}_2(\text{mbbp})_2](\text{pic})_2\)\(\text{DMF-CH}_3\text{OH}\) |
|---------|--------------------------------------------------|
| Molecular formula | \(\text{C}_{58}\text{H}_{49}\text{Ag}_{2}\text{N}_{17}\text{O}_{16}\) |
| Molecular weight | 1455.88 |
| Crystal system | Triclinic |
| Space group | \(P\)-1 |
| \(a\) (Å) | 13.4409(3) |
| \(b\) (Å) | 14.6508(3) |
| \(c\) (Å) | 15.1077(3) |
| \(\alpha\) (°) | 83.6370(10) |
| \(\beta\) (°) | 76.3740(10) |
| \(\gamma\) (°) | 83.4160(10) |
| \(V\) (Å\(^3\)) | 2861.36(10) |
| \(Z\) | 2 |
| \(\rho_{\text{calc}}\) (mg m\(^{-3}\)) | 1.690 |
| Absorption coefficient (mm\(^{-1}\)) | 0.774 |
| \(F\) (000) | 1476.0 |
| Crystal size (mm) | 0.39 × 0.33 × 0.24 |
| Radiation | MoK\(\alpha\) (\(\lambda\) = 0.71073) |
| 2\(\theta\) range for data collection (°) | 6.018–54.968 |
| Index ranges | \(-15 \leq h \leq 17, -19 \leq k \leq 18, -19 \leq l \leq 17\) |
| Reflections collected | 27,186 |
| Independent reflections | 12,774 \([R_{\text{int}} = 0.0174, R_{\text{sigma}} = 0.0238]\) |
| Data/restraints/parameters | 12,774/29/860 |
| Goodness-of-fit on \(F^2\) | 1.064 |
| Final \(R\), \(wR\) indices \([I > 2\sigma(I)]\) | \(R = 0.0328, wR_2 = 0.0859\) |
| \(R\), \(wR\) indices (all data) | \(R = 0.0382, wR_2 = 0.0913\) |
| Largest peak and hole differences (e Å\(^3\)) | 1.34/−0.92 |
following method: To a stirred solution of mbbp (0.1358 g, 0.40 mmol) in hot MeOH (10 mL), Ag(pic) (0.0672 g, 0.20 mmol) was added and dissolved in MeOH (5 mL) and CH₂Cl₂ (5 mL). A pale-yellow precipitate was generated immediately. The sediment was filtered off, washed with absolute ethanol, and dried in vacuo. The dried precipitate was dissolved in DMF to give a yellow solution, and yellow crystals suitable for X-ray diffraction studies were obtained by evaporation. Yield: 54%. Elemental analysis for C₅₄H₃₈Ag₂N₁₆O₁₄: calculated (%): C, 48.02; H, 2.84; N, 16.59; O, 16.58. Found (%): C, 48.07; H, 2.80; N, 16.36; O, 16.62. Selected IR data (KBr ν/cm⁻¹): 1605 ν(C=C), 1473 ν(C=N), 1263 ν(C–N). UV/Vis (DMF): λ = 325, 380 nm.

X-ray crystallography

The intensity data of the complex were collected on a Bruker APEX II area detector with graphite-monochromated MoKα radiation (λ = 0.71073 Å) at 153 K. Data reduction and cell refinements were performed using the SMART and SAINT programs. Absorption corrections were carried out by the empirical method. The structure was solved and refined by direct methods and refined by full-matrix least-squares against F² using OLEX 2 software. All H atoms were found in different electron maps and were subsequently refined in a riding-model approximation with C–H distances ranging from 0.95 to 0.99 Å. Information concerning the crystallographic data collection and structural refinements is summarized in Table 2. The relevant bond lengths and angles are listed in Table 3. The crystallographic data (excluding structure factors) for complex in this paper have been deposited with the Cambridge Crystallographic Data Centre as CCDC 2008542. Copies of the data can be obtained free of charge on application to the CCDC, 12 Union Road, Cambridge CB2 1EZ, UK.

Declaration of conflicting interests

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Supplemental material

Supplemental material for this paper is available online.

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| Table 3. Selected bond lengths (Å) and angles (°) for the Ag(I) complex. |
|-------------------------------------------------|
| Bond length                                       |
| Ag(1)–Ag(2) 2.991(3)                             |
| Ag(1)–N(10) 2.752(2)                            |
| Ag(2)–N(3) 2.126(2)                             |
| Ag(2)–N(5) 2.692(2)                             |
| Bond angle                                      |
| N(1)–Ag(1)–Ag(2) 86.13(5)                      |
| N(10)–Ag(1)–Ag(2) 55.83(4)                     |
| N(3)–Ag(2)–N(8) 179.52(9)                      |
| N(1)–Ag(1)–N(10) 109.14(7)                     |
| N(3)–Ag(2)–Ag(1) 92.28(6)                      |
| N(2)–Ag(2)–Ag(1) 87.61(5)                      |
| Ag(1)–N(1) 2.131(2)                             |
| Ag(1)–N(6) 2.126(2)                             |
| Ag(2)–N(8) 2.126(19)                            |
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