

A Double-Helical Silver(I) Coordination Polymer Based on 1-(4-pyridylmethyl)-1*H*-benzotriazole: Synthesis, Crystal Structure and Luminescent Property

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Abstract: The reaction of AgNO₃ with 1-(4-pyridylmethyl)-1*H*-benzotriazole (4-pbt) afforded a photoluminescent one-dimensional (1D) double-helical coordination polymer {[Ag(4-pbt)](NO₃)}_∞ (**1**) formed by interchain C-H...π supramolecular interactions of the single-helical chains. Adjacent 1D double-helical motifs aforementioned were assembled into different two-dimensional (2D) sheets from the different crystallographic directions, respectively, by the co-effects of Ag...O, C-H...O H-bonding, π...π stacking, and C-H...π supramolecular interactions. In comparison with the previous finding, the present work reveals that the N-donor spatial position of pendant pyridyl group in such benzotriazol-1-yl-based pyridyl ligands plays an important role on the final structures of relevant coordination complexes.

Keywords: Silver(I) coordination polymer, crystal structure, double helix, luminescence.

INTRODUCTION

The rational engineering and controlled preparation of coordination architectures is currently of great interest in coordination and supramolecular chemistry because of their interesting topologies [1-6] and potential uses as functional materials [7-11]. In this field, linked historically to the introduction of the first inorganic double-stranded helicate by Lehn *et al.* [12], a number of artificial helical supramolecular assemblies based on different predesigned ligands have been extensively focused and well reported due to both their aesthetically appealing topologies and their potential applications, such as asymmetric catalysis and nonlinear optical materials [13-20]. One of the most successful strategies for constructing such complexes has been the assembly reaction of different metal ions (as nodes) with well-designed organic ligands (as building blocks), which, so far, has been at an evolutionary stage with the current focus mainly on understanding the factors to determine the crystal packing as well as exploring relevant potential properties [1-11].

Among various ligands, numerous related bis-heterocyclic chelating or bridging ligands have been synthesized and extensively been used to construct functional coordination complexes, which contain different hetero-aromatic ring systems, for example pyridine, pyrazine, quinoline, quinoxaline, pyrazole, imidazole, thiazoles, and their benzo-analogues [21-23]. As such, we also found that Richardson and Steel have initially reported the studies concerning five N-containing bis-heterocyclic ligands bearing 1-substituted benzotriazole subunits, and their Ru^{II}, Cu^{II}, Pd^{II}, and Ag^I complexes [24]. To further investigate the influences of the N-donor spatial position of pendant pyridyl group in struc-

turally related benzotriazol-1-yl-based pyridyl ligands on the structures of their coordination complexes, a new N-containing heterocyclic ligand 1-(4-pyridylmethyl)-1*H*-benzotriazole (4-pbt, see Chart 1) was designed and prepared, and its reaction with AgNO₃ offered a 1D double-helical coordination polymer {[Ag(4-pbt)](NO₃)}_∞ (**1**) formed by C-H...π supramolecular interactions between adjacent single-helical chains. The relevant result reveals that the N-donor spatial position of pendant pyridyl group in such benzotriazol-1-yl-based pyridyl ligands, in comparison with the previous finding [24], plays an important role on the final structures of relevant coordination complexes. We herein reported the synthesis, crystal structure, and luminescent property of complex **1**.

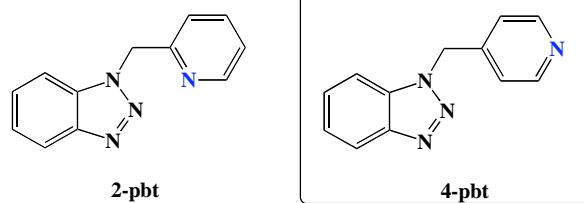


Chart 1.

EXPERIMENTAL

Materials and General Methods

All the reagents and solvents for synthesis were commercially available and used as received without further purification. Melting points were measured on an X-4 micro melting point detector without further correction. Elemental analyses (C, H, N) were performed on a Perkin-Elmer 240C analyzer. The IR spectra were recorded in the range 4000-400 cm⁻¹ on a Tensor 27 OPUS (Bruker) FT-IR spectrometer with KBr pellets. ¹H NMR spectra were recorded on a Bruker AC-

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P500 spectrometer (300 MHz) at 25 °C in CDCl₃ with tetramethylsilane as the internal reference. The emission/excitation spectra were recorded on a JOBIN YVON (HORIBA) FLUOROMAX-P spectrophotometer at room temperature.

Synthesis of Ligand

1-(4-Pyridylmethyl)-1*H*-benzotriazole (4-pbt) was synthesized according to the modified literature procedure [25]. Benzotriazole (0.26 g, 2.2 mmol), 4-(chloromethyl)pyridine hydrochloride (4-picoyl chloride hydrochloride) (0.33 g, 2 mmol), and potassium carbonate (1.52g, 11 mmol) were put in suspension in 50 mL of CH₃CN. The mixture was stirred at ambient temperature of the laboratory for *ca.* 1 h before being heated at reflux over 24 h with vigorous stirring. The remaining beige precipitate was filtered off and rinsed with CH₃CN. The solvent was removed from the filtrate, and the beige product was taken up in CHCl₃ and washed three times with H₂O before being dried over anhydrous MgSO₄. 1-(4-Pyridylmethyl)-1*H*-benzotriazole (4-pbt) ligand was obtained as yellow powder and purified by recrystallization from CHCl₃/hexane (Yield: ~40% based on 4-(chloromethyl)pyridine hydrochloride). Mp: 111~113 °C. Elemental analysis (%) Calcd. for C₁₂H₁₀N₄, *M* = 210.24: C, 68.56; H, 4.79; N, 26.65. Found (%): C, 68.47; H, 4.85; N, 26.54. IR (KBr pellet, cm⁻¹): 3032(w), 2960(w), 1678(w), 1599(s), 1560(w), 1495(m), 1454(w), 1419(vs), 1374(w), 1317(m), 1263(m), 1225(s), 1160(m), 1097(s), 1069(w), 992(w), 948(w), 910(w), 845(w), 798(s), 764(m), 739(vs), 664(w), 583(w), 524(w), 480(m), 459(w). ¹H NMR (300 MHz, CDCl₃, 25 °C, TMS): δ 5.876 (s, 2H), 7.107 (d, *J* = 5.4 Hz, 2H), 7.270-7.477 (M, 3H), 8.122 (d, *J* = 8.1 Hz, 1H), 8.580-8.599 (m, 2H).

Synthesis of Complex 1

A solution of 4-pbt (0.05 mmol) in CH₃OH (10 mL) was carefully layered on top of a H₂O solution (15 mL) of AgNO₃ (0.1 mmol) in a test tube. Light yellow single crystals suitable for X-ray analysis appeared at the tube wall after *ca.* one month at room temperature. Yield: ~30% based on 4-pbt. Elemental analysis (%) Calcd. for C₁₂H₁₀AgN₅O₃, *M* = 380.12: C, 37.92; H, 2.65; N, 18.42. Found (%): C, 37.79; H, 2.71; N, 18.51. IR (KBr pellet, cm⁻¹): 3035(w), 1946(w), 1767(w), 1606(m), 1562(w), 1497(w), 1455(w), 1358(w), 1226(s), 1164(m), 1099(w), 1072(w), 1014(w), 958(w), 854(w), 823(m), 741(s), 666(w), 624(m), 560(m), 483(w).

X-Ray Crystallographic Studies of Complex 1

X-ray single-crystal diffraction measurements for complex **1** were carried out on a Bruker Smart 1000 CCD area-detector diffractometer equipped with a graphite crystal monochromator situated in the incident beam for data collection at 293(2) K. The determinations of unit cell parameters and data collections were performed with Mo-K α radiation ($\lambda = 0.71073$ Å) by the ω scan mode. There was no evidence of crystal decay during data collection. The program SAINT [26] was used for integration of the diffraction profiles. Semi-empirical absorption corrections were applied using SADABS program [27]. The structure was solved by a direct method using the SHELXS program of the SHELXTL package and refined with SHELXL [28]. Metal atoms in complex **1** were located from the *E*-maps, and other non-hydrogen

atoms were located in successive difference Fourier syntheses and refined with anisotropic thermal parameters on F^2 . The hydrogen atoms were added theoretically, riding on the concerned atoms and refined with fixed thermal factors. Crystallographic data and experimental details for structural analyses are listed in Table 1 and H-bonding geometries are listed in Table 2. CCDC No. 685715 contains the supplementary crystallographic data for complex **1**. This material can be obtained free of charge via <http://www.ccdc.cam.ac.uk/deposit>, or from the Cambridge Crystallographic Data Centre, 12 Union Road, Cambridge CB2 1EZ, UK; fax: (+44) 1223-336-033; or e-mail: deposit@ccdc.cam.ac.uk.

Table 1. Crystallographic Data and Structure Refinement Parameters for Complex 1

	1
Empirical formula	C ₁₂ H ₁₀ AgN ₅ O ₃
Formula weight	380.12
Crystal system	Monoclinic
Space group	<i>C2/c</i>
Unit cell dimensions (Å, °)	
<i>a</i>	19.960(4)
<i>b</i>	8.6785(17)
<i>c</i>	18.633(4)
α	90
β	121.34(3)
γ	90
Volume (Å ³)	2756.7(12)
<i>Z</i>	8
<i>D</i> _{calcd} (g/cm ³)	1.832
μ (mm ⁻¹)	1.480
Crystal size (mm)	0.18 × 0.12 × 0.09
<i>F</i> (000)	1504
Range of <i>h, k, l</i>	-23/23, -10/10, -22/21
Reflections collected/unique	13318/2424
Max. & min. transmission	0.8783 & 0.7765
Data/restraints/parameters	2424/12/190
<i>R</i> (int)	0.0273
Goodness-of-fit on F^2	1.118
<i>R</i> ₁ ^a & <i>wR</i> ₂ ^b [<i>I</i> > 2 σ (<i>I</i>)]	0.0548 & 0.1363
<i>R</i> ₁ ^a & <i>wR</i> ₂ ^b (all data)	0.0577 & 0.1386
Largest diff. peak & hole (e. Å ⁻³)	1.228 & -0.970

^a $R_1 = \sum(|F_o| - |F_c|)/\sum|F_o|$; ^b $wR_2 = [\sum w(|F_o|^2 - |F_c|^2)^2/\sum w(F_o^2)]^{1/2}$, where F_o = observed and F_c = calculated structure factors, respectively.

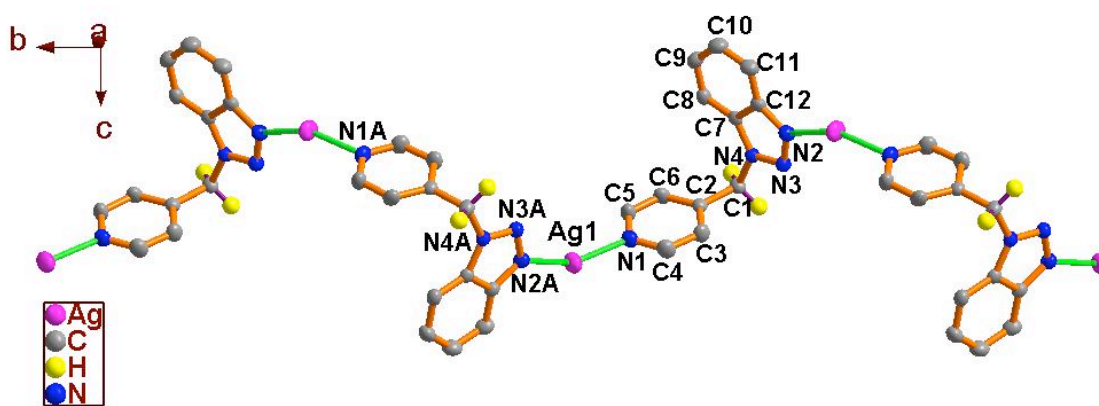


Fig. (1). ORTEP view of complex **1** with 30% thermal ellipsoid probability, showing coordination environment of Ag^{I} in the helical cationic chain [Atoms labeled with the suffix A are generated by the symmetry operation $(-x + 1, y + 1, -z + 3/2)$; H atoms except methylene spacer and NO_3^- anions were omitted for clarity].

X-Ray Powder Diffraction Studies of Complex 1

The X-ray powder diffraction patterns (XRPD) of **1** were recorded on a Rigaku D/Max-2500 diffractometer, operated at 40 kV and 100 mA, using a Cu-target tube and a graphite monochromator. The intensity data were recorded by continuous scan in a $2\theta/\theta$ mode from 3° to 80° with a step size of 0.02° and a scan speed of 8° min^{-1} . Simulation of the XRPD spectra was carried out by the single-crystal data and diffraction-crystal module of the *Mercury* (Hg) program available free of charge via the Internet at <http://www.iucr.org>.

RESULTS AND DISCUSSION

General Characterizations

Complex **1** is air stable. In general, the IR spectra show features attributable to each component of the complex [29]. The IR spectra for **1** show absorption bands resulting from the skeletal vibrations of the aromatic rings in $1600\text{--}1400 \text{ cm}^{-1}$ region as well as the characteristic band of NO_3^- anion at 1358 cm^{-1} . It should be noted that, due to the coordination of the pyridyl ring of 4-pbt ligand, the strong absorption band at $\sim 1575 \text{ cm}^{-1}$ resulting from the skeletal vibrations of the aromatic rings of the free ligand shifted to 1606 cm^{-1} in **1**, which were in good agreement with their solid structural features from the results of crystal structures [30]. As such, the elemental analysis of **1** is also consistent with the result of its structural analysis.

Crystal Structure of Complex 1

Single crystal X-ray diffraction analysis reveals that the crystal structure of complex **1** consists of NO_3^- anions and 1D helical cationic chains $\{[\text{Ag}(4\text{-pbt})]^+\}_\infty$ (Fig. 1-3). As depicted in Fig. (1), in the helical cationic chain, there is only one crystallographic independent Ag^{I} center that is two-coordinated by two N-atom donors, one N donor being from benzotriazole ring of one 4-pbt ligand and the other being from pyridyl ring of another different 4-pbt ligand. The Ag-N bond distances [$\text{Ag1-N1} = 2.186(5) \text{ \AA}$ and $\text{Ag1-N2} = 2.163(4) \text{ \AA}$] and the N1-Ag1-N2 angle of $152.8(2)^\circ$ are within the expected range for such complexes [31]. Each 4-pbt ligand takes a μ_2 -bridging coordination mode to connect

two Ag^{I} centers, generating a left-handed single-helical chain interwinded with a period of $17.357(3) \text{ \AA}$. In each 4-pbt of complex **1**, the dihedral angle between pyridyl and benzotriazole rings is $84.0(3)^\circ$. The single-helical chains were further assembled into a 1D infinite double-helical motif by the in-chain C-H $\cdots\pi$ supramolecular interactions involving the C7~C12 benzene rings (centroid $Cg1$) and the N2~N4/C7/C12 triazole rings (centroid $Cg2$) of 4-pbt ligands with an edge-to-face orientation [$d = 3.05$ and 3.20 \AA and $A = 146$ and 134° in the C-H $\cdots\pi$ patterns; d and A stand for the H $\cdots Cg$ separations and C-H $\cdots Cg$ angles in the C-H $\cdots\pi$ patterns, respectively] (see Fig. 2 and Table 2) [32]. Within the double-helical motif, the closest interchain Ag \cdots Ag non-bonding separation is $4.5371(1) \text{ \AA}$, which is obviously longer than that (2.889 \AA) found in metallic silver [33,34] and than those found in other complexes containing Ag \cdots Ag close contact (the van der Waals Ag \cdots Ag contact distance being 3.40 \AA) [35,36]. Besides, there were no obvious existences of inter-chain $\pi\cdots\pi$ stacking interactions between the pyridyl and benzotriazole ring of 4-pbt ligands within the 1D double-helical motif of **1** (the closest centroid-centroid separation is 4.565 \AA checked and calculated by PLATON procedure [37], which is much longer than 3.8 \AA) [38].

Furthermore, the adjacent 1D double-helical motifs were linked together to form the different 2D sheets from the different crystallographic directions (see Fig. 3), and then a overall 3D supramolecular network by the co-effects of a series of noncovalent interactions, respectively, such as $\pi\cdots\pi$ stacking interactions between completely parallel benzotriazole rings of adjacent 4-pbt ligands (centroid-centroid separation: 3.6067 \AA ; average interplanar separation: 3.4387 \AA ; see Fig. 3a), Ag \cdots O weak interactions [$\text{Ag1}\cdots\text{O1}$: $2.6932(2) \text{ \AA}$; $\text{Ag1}\cdots\text{O2}$: $2.7975(6) \text{ \AA}$], C-H \cdots O H-bonding interactions [$\text{C11-H11A}\cdots\text{O1}$ and $\text{C1-H1A}\cdots\text{O3}$, see Fig. (3b) and Table 2 for details], and C-H $\cdots\pi$ supramolecular interactions involving the C2~C6/N1 pyridyl rings (centroid $Cg3$) [$\text{C10-H10A}\cdots Cg3$, see also Table 2 for details] [32]. Therefore, various intra- or inter-molecular weak interactions mentioned above play an important role in the formation of **1**, especially in the aspect of linking the low-dimensional entities into higher-dimensional supramolecular network.

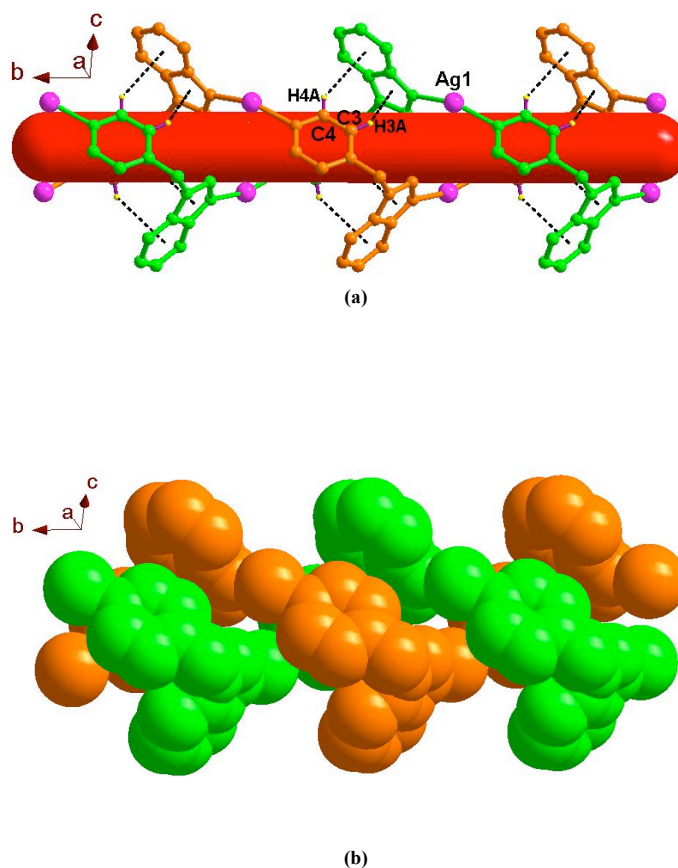


Fig. (2). View of (a) the 1D double-helical chain in **1** formed by the interchain C-H... π interactions between the pyridyl and benzotriazole rings (black dashed lines) and (b) the space-filling representation of the double-helical chain down the direction normal to helical axis (Only H atoms involved in the C-H... π interactions were shown for clarity).

Table 2. Hydrogen-Bonding Geometry (\AA , $^\circ$) for Complex **1^{a,b}**

D-H...A	$d(\text{D-H})$	$d(\text{H...A})$	$d(\text{D...A})$	D-H...A
C4-H4A...Cg1 ⁱ	0.930	3.05	3.862(7)	146
C3-H3A...Cg2 ^j	0.930	3.20	3.901(4)	134
C11-H11A...O1 ^j	0.930	2.48	3.225(2)	137
C1-H1A...O3 ^k	0.970	2.41	3.369(2)	170
C10-H10A...Cg3 ^l	0.930	2.97	3.755(7)	143

^a Symmetry code for **1**: (i): $-x + 1, y, -z + 3/2$; (j): $-x + 1, -y + 1, -z + 2$; (k): $x, -y + 1, z - 1/2$; (l): $x, -y + 2, z + 1/2$.

^b Cg1, Cg2, and Cg3 are the centroids of the C7-C12 benzene rings, the N2-N4/C7/C12 triazole rings, and the C2-C6/N1 pyridyl rings of 4-pbt ligands, respectively.

Richardson and Steel [24] have initially synthesized a 1-substituted benzotriazol-1-yl-based pyridyl ligand, 1-(2-pyridylmethyl)-1*H*-benzotriazole (2-pbt, see also Chart 1). The result indicates that the silver nitrate complex of 2-pbt is a cyclic Ag₂(2-pbt)₂ dimer in which 2-pbt coordinates *via* the pyridyl and N3 donor of benzotriazole jointly to bridge two Ag^I centers with non-bonding Ag...Ag separation of 4.942(1) \AA in a dinuclear unit. However, by using 1-(4-pyridylmethyl)-1*H*-benzotriazole (4-pbt) instead of 2-pbt to react with AgNO₃ under conventional solution condition, we have isolated a 1D double-helical coordination polymer {[Ag(4-pbt)](NO₃)}_∞ (**1**) formed by interchain C-H... π su-

pramolecular interactions of the single-helical chains. Thus, the different spatial position of coordinated N donors in the pendant pyridyl groups of 4-pbt and 2-pbt ligands affected the final crystal structures of their metal-organic complexes. This fact may offer the means to construct coordination architecture with potential properties just by variations of N-donor spatial position of such N-containing hetero-aromatic ligands.

Luminescent Property

Luminescent supramolecular architectures are currently of great interest because of their potential applications in

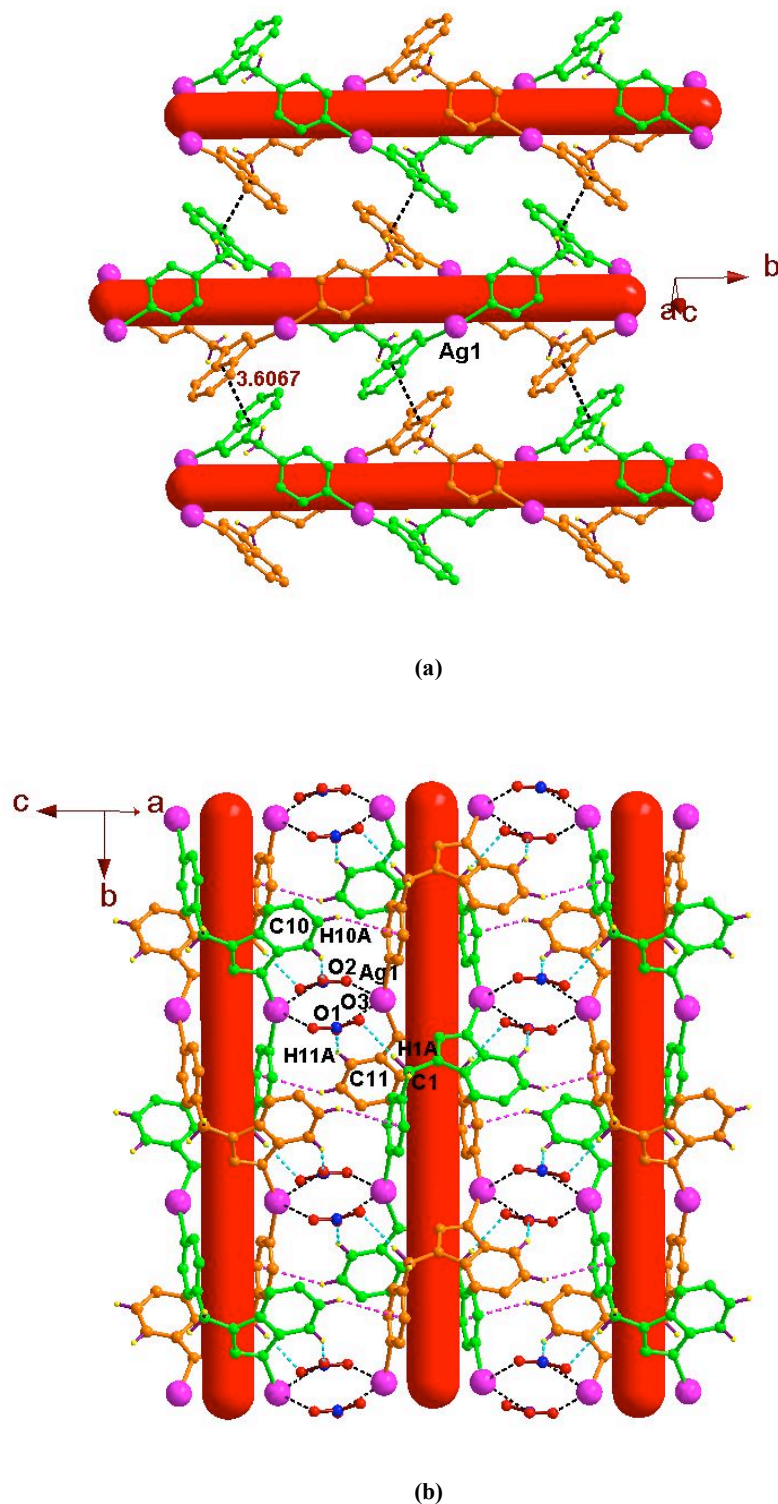


Fig. (3). View of (a) the 2D network in **1** formed by interchain $\pi\cdots\pi$ stacking interactions (black dashed lines) between the benzotriazole rings of 4-pbt ligands and (b) the 2D network in **1** formed by the co-effects of the interchain C-H $\cdots\pi$ (purple dashed lines), C-H \cdots O (cyan dashed lines), and Ag \cdots O (black dashed lines) interactions.

optoelectronic devices or as fluorescent sensors and probes [5,36]. Complex **1** exhibits an intense emission band at $\lambda_{\text{max}} = 539$ nm upon excitation at 311 nm in the blue region (see Fig. 4), while the free 4-pbt ligand displays the luminescence in the solid state at $\lambda_{\text{max}} = 342$ nm upon excitation at $\lambda = 307$

nm. Complex **1** has electron rich d^{10} metal Ag^I and low-lying $p\pi^*$ acceptor orbitals of 4-pbt ligand, therefore, the possible origins of the emission in **1** may be tentatively assigned to be MLCT (metal-to-ligand charge transfer) from silver (4d) to the 4-pbt ligand (π^*) [15,39,40]. In general, Ag^I coordination

polymers display weak luminescence at room temperature [5,36]. They are known to be greater emitting materials at low temperature with an enhancement of the emitting intensity and/or a shift of the emission wavelength. Therefore, our structure of **1** represents one new example of the room-temperature luminescent Ag^I-containing coordination polymer.

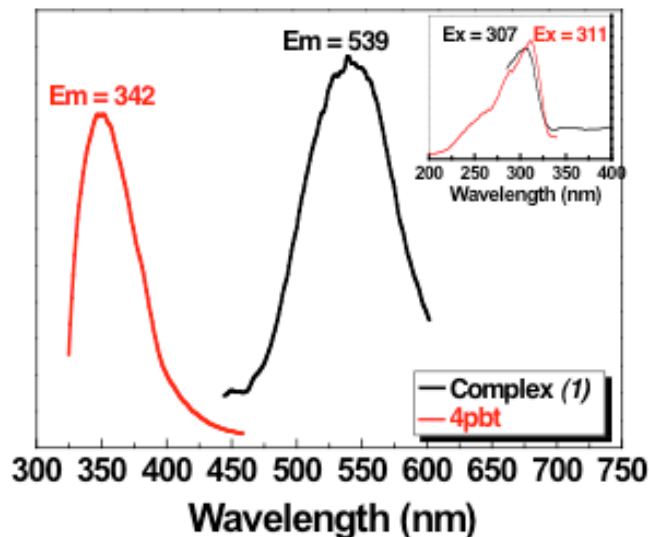


Fig. (4). Solid emission and excitation (Inset at the top right corner) spectra of **1** at room temperature.

XRPD Results

To confirm whether the crystal structures are truly representative of the bulk materials for related luminescent measurement of complex **1**, X-ray powder diffraction (XRPD) experiments have been carried out for **1**. The XRPD experimental and computer-simulated patterns of the corresponding complexes are shown in Fig. (5). Although the experimental patterns have a few unindexed diffraction lines and some are slightly broadened in comparison with those simulated from the single crystal modes, it still can be considered favorably that the bulk synthesized materials and the as-grown crystals are homogeneous for **1**.

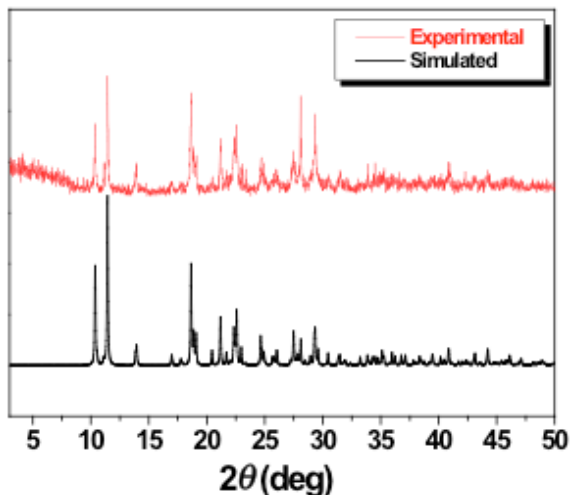


Fig. (5). X-ray powder diffraction (XRPD) pattern for complex **1**.

CONCLUSION

In conclusion, a novel Ag^I coordination polymer having infinite 1D double-helical chain structure has been successfully constructed by using an achiral N-containing heterocyclic aromatic ligand 1-(4-pyridylmethyl)-1H-benzotriazole (4-pbt). The result reveals that, in comparison with the previous finding, the N-donor spatial position of pendant pyridyl group in such benzotriazol-1-yl-based pyridyl ligands plays an important role on the final structure of relevant coordination complex. The solid emission exhibited a favorable luminescent property at room temperature resulting from the metal-to-ligand charge transfer (MLCT).

ACKNOWLEDGEMENTS

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