Novel Linking Ligand Containing Sulfur-Donor Atoms and Its Compounds of Palladium and Silver

Hee K. Lee and Soon W. Lee[*]

Department of Chemistry (BK21), Institute of Basic Science, Sungkyunkwan University, Natural Science Campus,
Suwon 440-746, Korea. [*]E-mail: swlee@chem.skku.ac.kr
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A linking ligand containing sulfur donor atoms in the terminal thiophene rings, 1,2-bis(thiophen-2-ylmethylene)hydrazine (**L**), was prepared by Schiff-base condensation. Ligand **L** reacted with [PdCl$_2$(NCPh)$_2$] to produce a molecular Pd compound [Pd**L**$_2$Cl$_2$] (**1**). On the other hand, it reacted with AgNO$_3$ and AgClO$_4$ to produce a 2-D network [Ag**L**$_{0.5}$(NO$_3$)] (**2**) and a 1-D polymer [Ag**L**]ClO$_4$ (**3**), respectively, whose structures are based on secondary intermolecular forces such as H-bonding, van der Waals interaction, and π-π stacking. Polymer **2** exhibited photoluminescence at room temperature in the solid state.

Key Words : Linking ligand containing sulfur donor atoms, Silver coordination polymers

Introduction

Coordination polymers with various channels or cavities have useful properties applicable to catalysis, chirality, conductivity, luminescence, magnetism, adsorption, porosity, and gas storage, and therefore they are currently under intensive study.[1-4] For the preparation of these polymers, multifunctional linking ligands such as pyridyls and carboxylates with strongly coordinating hard oxygen- or nitrogen-donor atoms are typically used.[5-10]

There have recently been a series of papers demonstrating that secondary weak interactions, which include hydrogen bonding, van der Waals interaction, electrostatic interaction, and π-π stacking, are of great importance in constructing complex or gigantic compounds.[11-14] For example, DNA retains its double helical structure with the hydrogen bonding, one of such interactions. Novel coordination polymers of various types are currently prepared by self-assembly utilizing these secondary interactions, and this type of activity is expected to continue further in the future.[15-17]

We have recently prepared several long dipyridyl-type linking ligands by Schiff-base condensation (Chart 1).[18-20] We decided to expand this methodology to synthesize novel linking ligands that contains soft sulfur-donor atoms. In particular, we have focused on silver coordination polymers of this type of ligands, due to the fact that the study on silver

coordination polymers of linking ligands containing hetero-aromatic rings with sulfur atoms has been relatively unexplored.[21] In addition, it recently appeared in the literature that luminescent properties of silver coordination polymers are different from those of cationic silver compounds.[22-27] In this study, we prepared a novel linking ligand, 1,2-bis-(thiophen-2-ylmethylene)hydrazine (**L**), which contains terminal heteroaromatic rings with sulfur atoms (Chart 1). Herein, we report the preparation of this ligand and its reactions with Pd and Ag complexes, together with luminescent properties of one Ag coordination polymer.

Experimental Section

All chemicals were purchased and purified. All solvents were distilled and stored over molecular sieves. Experiments involving silver compounds were performed in the dark because silver compounds are potentially light-sensitive. IR spectra were recorded with a Nicolet 320 FTIR spectrophotometer. Elemental analyses were performed with EA1110 (CE instrument, Italy) at the Korea Basic Science Institute. Luminescent experiments were conducted on a TA4000/SDT 2960 instrument. [PdCl$_2$(NCPh)$_2$] was prepared by the literature method.[28]

Preparation of 1,2-bis(thiophen-2-ylmethylene)hydrazine (L). To an ethanol solution (30 mL) containing hydrazine (35 wt% solution in H$_2$O) (0.916 mL, 10 mmol) and 2-thiophenecarboxaldehyde (1.836 g, 20 mmol) was added a dichloromethane solution (30 mL) containing formic acid (0.2 mL), and the solution was stirred for 4 h. The resulting brown slurry was filtered, washed with hexane (25 mL × 2), and dried under vacuum to give a yellow solid of **L**. 97% yield. mp: 136-138 °C. IR (KBr, cm^{-1}): 1607, 1401, 718. Anal. Calcd for C$_{10}$H$_8$N$_2$S$_2$ (M_r = 222.30): C, 54.02; H, 4.53; N, 12.60; S, 28.84. Found: C, 53.84; H, 4.87; N, 12.86; S, 28.68.

Preparation of [PdL$_2$Cl$_2$] (1). At room temperature, a benzene solution (3 mL) containing [PdCl$_2$(NCPh)$_2$] (0.055
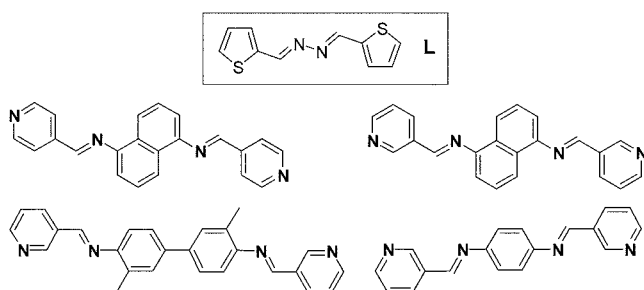

Chart 1

g, 0.14 mmol) was layered onto the top of a dichloromethane solution (3 mL) containing **L** (0.035 g, 0.16 mmol) in a test tube. After 3 days, the resulting solution was filtered, washed with diethyl ether (25 mL × 2), and then air-dried to give yellow needle crystals of **1**. 60% yield. mp: 180-182 ℃ (decom). IR (KBr, cm^{-1}): 3100, 1711, 1291, 703. Anal. Calcd for $C_{20}H_{16}ClN_4S_4Pd$ (M_r = 617.91): C, 38.88; H, 2.61; N, 9.07. Found: C, 39.05; H, 2.32; N, 8.80.

Preparation of [AgL$_{0.5}$(NO$_3$)] (2). At room temperature, an acetonitrile solution (3 mL) containing AgNO$_3$ (0.015 mg, 0.08 mmol) was layered onto the top of a dichloromethane solution (3 mL) containing **L** (0.019 g, 0.09 mmol). After 2 days, the resulting solution was filtered, washed with diethyl ether (25 mL × 2), and then air-dried to give yellow crystals of **2**. 71% yield. mp: 186-188 ℃ (decom). IR (KBr, cm^{-1}): 3089, 1605, 1391, 715. Anal. Calcd for $C_5H_3N_2O_3$-SAg (M_r = 279.02): C, 21.52; H, 1.08; N, 10.04. Found: C, 21.29; H, 1.13; N, 10.91.

Preparation of [AgL$_2$]ClO$_4$ (3). Polymer **3** was prepared in the same way as polymer **2**. 66% yield. mp: 218-220 ℃ (decom). IR (KBr, cm^{-1}): 3068, 1603, 1560, 1421, 1102,

713, 624. Anal. Calcd for $C_{20}H_{16}N_4O_4S_4ClAg$ (M_r = 647.93): C, 37.07; H, 2.49; N, 8.65. Found: C, 37.10; H, 2.15; N, 9.51.

X-ray Structure Determination. All X-ray data were collected with a Siemens P4 diffractometer equipped with a Mo X-ray tube. Intensity data were empirically corrected for absorption with ψ-scan data. All structures were solved by direct methods. All non-hydrogen atoms were refined anisotropically. Whereas hydrogen atoms in ligand **L** were located and refined isotropically, those in **1**-**3** were generated in idealized positions and refined in a riding model. All calculations were carried out with the SHELXTL programs.[29] Details on crystal data, intensity collection, and refinement details are given in Table 1. Selected bond lengths and angles are given in Table 2.

Crystallographic data for the structural analysis have been deposited at the Cambridge Crystallographic Data Center: 629389 (**L**), 629390 (**1**), 629391 (**2**), and 629392 (**3**). Copies of this information may be obtained free of charge from: The director, CCDC, 12 Union Road, Cambridge, CB2 1EZ, UK (Fax: +44-1223-336-033; e-mail: *deposit@ccdc.cam.ac.uk* or www: *http://www.ccdc.cam.ac.uk*).

Table 1. X-ray Data Collection and Structure Refinement Details

	L	**1**	**2**	**3**
formula	$C_{10}H_8N_2S_2$	$C_{20}H_{16}CLN_4S_4Pd$	$C_5H_3AgN_2O_3S$	$C_{20}H_{16}AgClN_4O_4S_4$
fw	220.30	617.91	279.02	647.93
temp, K	293(2)	293(2)	293(2)	293(2)
crystal size	0.32 × 0.25 × 0.08	0.58 × 0.18 × 0.10	0.38 × 0.14 × 0.10	0.44 × 0.24 × 0.16
color	yellow	yellow	yellow	yellow
cryst syst	monoclinic	monoclinic	triclinic	monoclinic
space group	$P2_1/n$	$P2_1/n$	$P\bar{1}$	$P2_1/n$
a, Å	9.785(2)	7.236(1)	4.0119(5)	8.471(1)
b, Å	11.411(1)	15.018(2)	9.0994(9)	12.442(1)
c, Å	9.861(2)	11.181(2)	10.786(1)	23.127(2)
α, deg			97.081(7)	
β, deg	100.78(1)	102.49(1)	99.82(1)	100.364(8)
γ, deg			102.257(8)	
V, Å^3	1081.6(3)	1186.3(3)	373.92(8)	2397.9(5)
Z	4	2	2	4
D_{calcd}, g cm^{-3}	1.353	1.730	2.478	1.795
μ, mm^{-1}	0.452	1.376	2.935	1.337
$F(000)$	456	616	268	1296
T_{min}	0.5312	0.7522	0.2396	0.5172
T_{max}	0.8537	0.9313	0.2773	0.8481
2θ range (°)	2.69-25.00	2.31-24.99	2.32-24.98	1.87-24.99
no. of reflns measured	1969	2238	1512	4472
no. of reflns unique	1853	2071	1307	4170
no. of reflns with $I > 2\sigma(I)$	1151	1822	1201	3235
no. of params refined	160	142	122	326
max. in $\Delta\rho$ (e Å^{-3})	0.142	0.327	0.658	0.404
min. in $\Delta\rho$ (e Å^{-3})	–0.164	–0.259	–0.547	–0.678
GOF on F^2	1.010	1.070	1.037	1.031
$R1^a$	0.0456	0.0489	0.0289	0.0456
$wR2^b$	0.0920	0.1100	0.0764	0.0990

$^aR1 = \Sigma||F_o| - |F||/\Sigma|F_o|$, $^bwR2 = \Sigma[w(F_o^2 - F_c^2)^2]/\Sigma[w(F_o^2)^2]^{1/2}$

Table 2. Selected Bond Lengths (Å) and Angles (deg) for **L**

		L			
N1-C5	1.281(4)	N1-N1#1	1.418(5)	C5-N1-N1#1	111.6(3)
		Compound **1**			
Pd1-N1	2.040(3)	Pd1-Cl1	2.301(1)	S1-C1	1.703(5)
S1-C4	1.720(4)	S2-C10	1.645(6)	S2-C7	1.682(4)
N1-C6	1.287(5)	N1#3-Pd1-N1	180.0	C6-N1-N2	112.1(3)
C5-N2-N1	115.4(3)				
		Polymer **2**			
Ag1-N1	2.305(3)	Ag1-O1	2.414(3)	Ag1-O1#4	2.523(4)
Ag1-O1#5	2.625(4)	Ag1-S1	2.941(1)	Ag1-S1#6	3.093(1)
N1-N1#8	1.414(6)	N1-Ag1-O1	159.1(1)	C1-S1-Ag1	158.2(2)
C4-S1-Ag1	90.9(1)	C1-S1-Ag1#7	116.2(2)		
		Polymer **3**			
Ag1-N1	2.136(6)	Ag1-N3	2.163(7)	Ag1-N3A	2.169(9)
N1-N2	1.317(7)	N3-N4	1.317(7)	C5-N1-Ag1	130.0(6)
N2-N1-Ag1	133.8(5)	C15-N3-Ag1	134.1(6)	N4-N3-Ag1	130.3(5)

Symmetry transformations used to generate equivalent atoms: #1 = $-x + 1$, $-y + 1$, $-z + 1$; #2 = $-x$, $-y$, $-z$; #3 = $-x$, $-y$, $-z + 1$; #4 = $-x$, $-y + 1$, $-z + 2$; #5 = $-x + 1$, $-y + 1$, $-z + 2$; #6 = $x - 1$, y, z; #7 = $x + 1$, y, z; #8 = $-x + 1$, $-y + 2$, $-z + 2$

Results and Discussion

Preparation of a ligand. A potential linking ligand (**L**) containing sulfur-donor atoms in two terminal heteroaromatic rings was prepared in high yield by the simple and straightforward Schiff-base condensation. Hydrazine reacted with 2-thiophenealdehyde to give **L** at room temperature under slightly acidic conditions (pH = 5-6) (eq. 1).


(1)
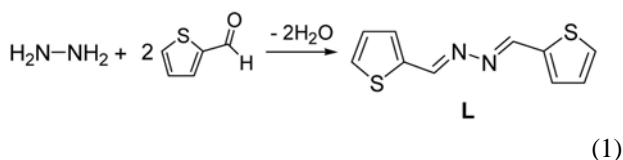
This ligand was characterized by elemental analysis, IR spectroscopy, and X-ray diffraction. Its IR spectra display an absorption band at 1607 cm^{-1} (**L**) assignable to the C=N bond, consistent with the fact that C=N stretching frequencies of Schiff bases appear in the range 1680-1603 cm^{-1}.[30-32] The structure of ligand **L** is shown in Figure 1. The central N-N bond length is 1.418(5) Å. The S···S distance between two terminal thiophene rings is 7.475 Å, and the whole molecule is conjugated.

Molecular palladium compound. We tried to prepare palladium coordination polymers by employing ligand **L**. For this purpose, PdCl$_2$(NCPh)$_2$ was selected, because it possesses labile benzonitrile (NCPh) ligands and a relatively soft Pd metal. Inconsistent with our expectation, however, this ligand produced a molecular Pd compound from the reaction with PdCl$_2$(NCPh)$_2$, in which the Pd metal is bonded to the nitrogen atoms in the Schiff-base imine groups, not sulfur atoms in the terminal heteroaromatic rings
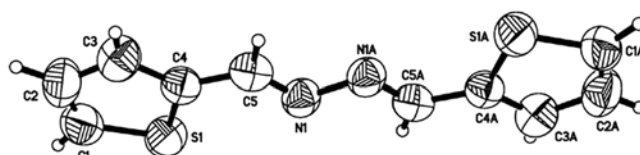

Figure 1. Molecular structure of **L** with 50% probability thermal ellipsoids.

(eq. 2).

(2)
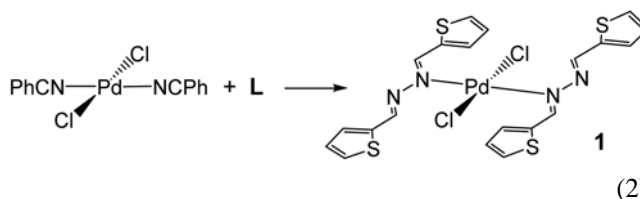
The molecular structure of compound **1** is given in Figure 2, which shows a square-planar coordination environment. Two terminal thiophene rings are twisted from each other with a dihedral angle of 9.4(2)°. The Pd-N bond length (2.036(4)) Å indicates a single Pd-N bond.

Silver coordination polymers. From the formation of the molecular Pd compound (**1**), rather than the Pd coordination polymer, we have come to a conclusion that the Pd-Cl and Pd-N bond strengths are considerably strong enough to preclude the formation of the target Pd coordination polymers. Therefore, we decided to modify synthetic strategies in such a way that the starting compound is replaced with a softer metal (Ag) compound containing labile ligands (NO$_3^-$, ClO$_4^-$). This modification allowed us to prepare a couple of silver coordination polymers based on secondary intermolecular forces. The 2-D silver coordination polymer [Ag**L**$_{0.5}$-
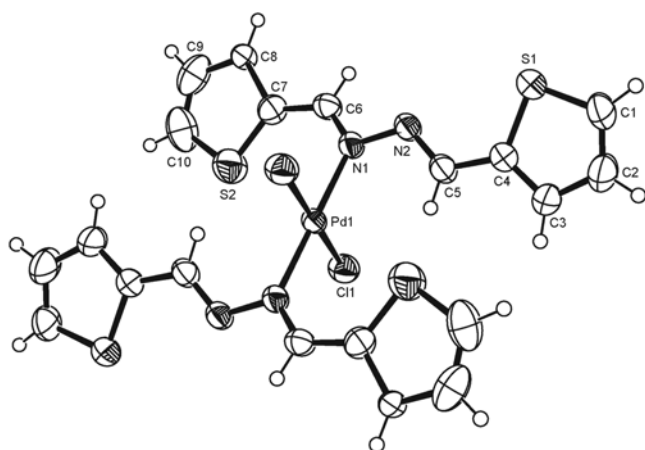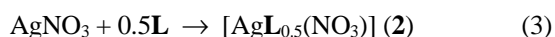
Figure 2. ORTEP drawing of compound **1**. Unlabeled atoms are related to labeled ones by the crystallographic inversion.
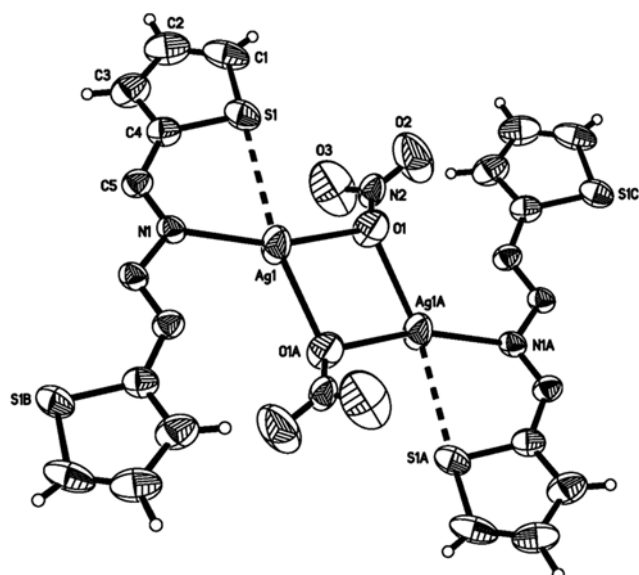

Figure 3. Local coordination environment of silver in polymer **2**.

(NO₃)] (**2**) was prepared by layering an acetonitrile solution containing AgNO₃ onto the top of a dichloromethane solution containing **L** at room temperature (eq. 3).

$$AgNO_3 + 0.5L \rightarrow [AgL_{0.5}(NO_3)] \ (2) \tag{3}$$

Polymer **2** shows several interesting structural aspects. Its asymmetric unit consists of one Ag, one half **L**, and one NO₃⁻ (Figure 3). Bond lengths (Ag-N = 2.305(3) Å; Ag-O = 2.414(3)-2.625(4) Å; Ag···S = 2.941(1)-3.093(1) Å) tell us that the Ag-N and Ag-O bonds are normal covalent bonds and the Ag···S bonds can be regarded as strong van der Waals contacts. In other words, the Ag metal is bound to one nitrogen and three oxygen atoms by normal covalent bonding and further connected to two sulfur atoms by van der Waals contacts of the type Ag···S. For comparison, the van der Waals radii of Ag and S atoms are 1.72 and 1.80 Å, respectively. In fact, these bonding parameters are inconsistent with our initial expectation that the soft silver metal forms a stronger bond with the softer sulfur atoms rather
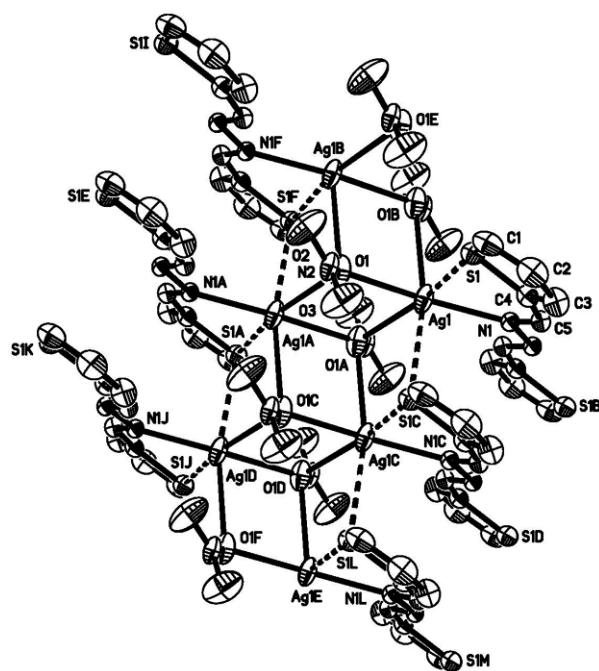

Figure 4. A chain of half-cubanes constructed by 6-coordinate silver ions.
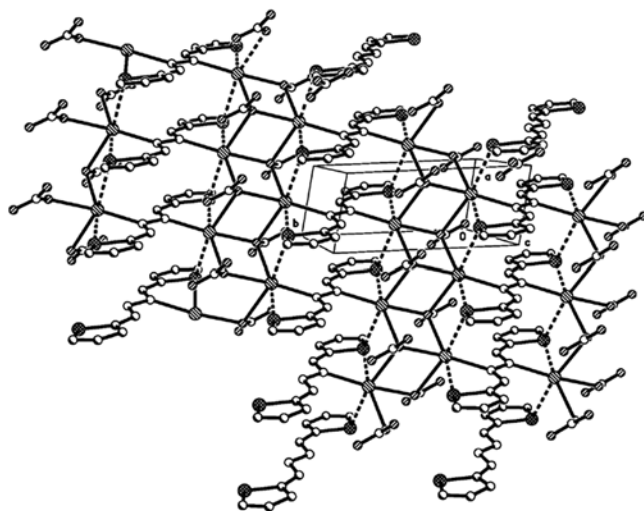
Figure 5. Packing diagram for **2**.

than the harder nitrogen atoms in **L**. Another intriguing structural aspect is the existence of an μ_3-O atom (O1) in the nitrato ligand that links three Ag metals.
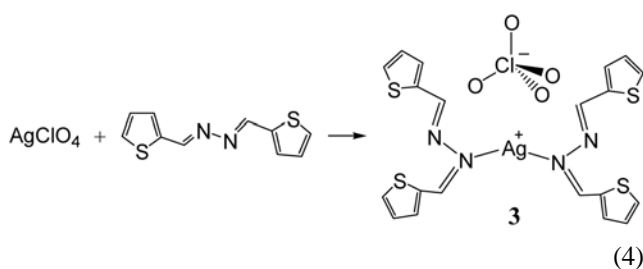
It is worth noting that silver and nitrate ions form a 1-dimensional stair in polymer **2** (solid lines in Figure 4). If van der Waals interactions between silver and sulfur atoms (dotted lines in Figure 4) are included, the silver ion can be regarded as 6-coordinate and the stair is expanded to a chain of incomplete (or half) cubanes. The half cubanes are linked by face-sharing to form an ultimate 2-D network. The packing diagram of polymer **2** along the *c*-axis is presented in Figure 5. Thiophene rings in ligands (**L**) lie in a specific orientation with a constant spacing, which results in π-π stacking. The spacing between the thiophene rings is 3.65 Å,
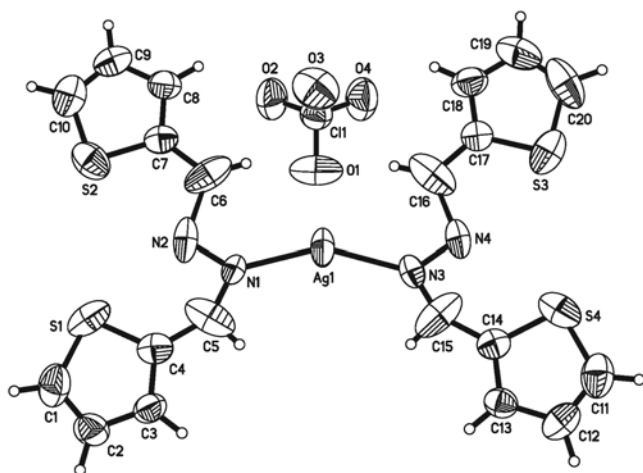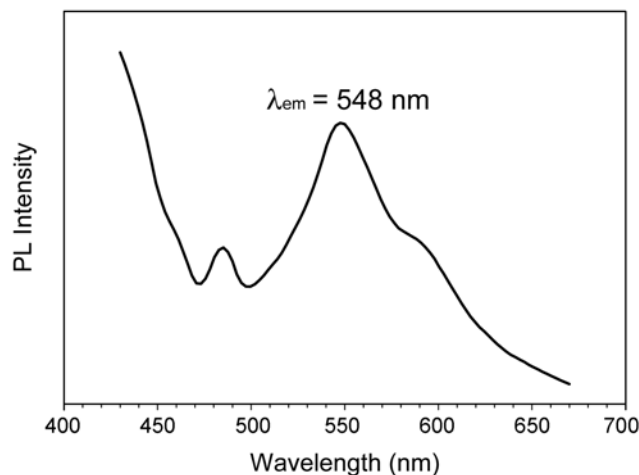
Figure 6. Monomer unit of polymer **3** showing the cis orientation of sulfur-donor atoms.

which is within a typical range for coordination polymers containing the π-π stacking.[33-35] The structure of polymer **2** clearly indicates that secondary interactions play a crucial role in determining the structural outcome of target coordination polymers.

Recently, it has been shown that some ionic silver coordination polymers exhibit photoluminescence that depends on the nature of the counterions in those ionic polymers[3,23,24] In this context, we tried to prepare other silver coordination polymers containing counterions other than nitrate. A 1-D silver coordination polymer [Ag**L**$_2$]ClO$_4$ (**3**) containing a perchlorate counterion was prepared from AgClO$_4$ and ligand **L** in the same way as that employed for polymer **2** (eq. 4). Unfortunately, the corresponding polymer containing a PF$_6^-$ counterion could not be prepared as X-ray quality crystals.

(4)

The structure of polymer **3** is presented in Figure 6. An interesting structural point is that the sulfur atoms in the thiophnene rings are now cis to each other. When ligand **L** is in the free state, its sulfur atoms are mutually trans (Figure 1). The *trans*-to-*cis* conversion of sulfur orientation on the formation of polymer **3** can probably be ascribed to van der Waals attractions between silver and sulfur atoms in neighboring molecules, which can be maximized in the *cis* orientation of the sulfur atoms. Figure 7 is a packing diagram of polymer **3**, which shows a 1-D chain based on van der Waals interactions. Unlike polymer **2**, polymer **3** contains 5-coordinate silver metals if Ag···Ag and Ag···S interactions are included (Ag···Ag = 3.375 Å; Ag···S = 3.378 or 3.397 Å)

Figure 7. Packing diagram of **3** showing van der Waals interactions (dashed lines).

Figure 8. Solid-state photoluminescent emission spectrum for polymer **2** at room temperature.

(Figure 7). Perchlorate (ClO$_4^-$) ions are not bound to silver metals and simply behave as counterions.

Luminescent property of polymer 2. Much research has recently focused on the luminescent properties of coordination polymers due to their potential applications as light-emitting diodes.[36-38] Polymer **2** exhibits photoluminescence in the range of 400-700 nm at room temperature in the solid state, whereas **L** and polymer **3** do not. Figure 8 shows the photoluminescent emission spectrum of polymer **2**, which displays several emission maximums at 455, 487, 548, and 589 nm (emission wavelength = 370 nm). The major peak at 548 nm results in a green emission. Silver coordination polymers are known to exhibit weak photoluminescence at around 480 nm at low temperature that depends on the nature of ligands,[3] and therefore the room-temperature photoluminescence of polymer **2** is somewhat interesting. It should be mentioned that Dong and co-workers have recently reported several silver coordination polymers exhibiting such luminescence.[25,26,39] In addition, Cheng's group pre-

pared a 3-D silver coordination polymer [$Ag_4(btcH)_2$] (btcH$_3$ = benzene-1,3,5-tricarboxylic acid) with strong blue luminescent properties.[40] The absence of luminescence for **L** and polymer **3**, which also contains ligand **L**, can exclude the possibility that the emission phenomenon is due to the intraligand transition. Unfortunately, however, we cannot provide a plausible explanation for the photoluminescence of polymer **2**.

In summary, we prepared a novel linking ligand, 1,2-bis(thiophen-2-ylmethylene)hydrazine (**L**), which contains sulfur-donor atoms. Ligand **L** reacted with $PdCl_2(NCPh)_2$ to produce a molecular Pd compound, [Pd**L**$_2$Cl$_2$] (**1**), in which the Pd metal is bound to the nitrogen atoms in the Schiff-base imine groups. By contrast, ligand **L** reacted with $AgNO_3$ and $AgClO_4$ to produce a 2-D network [Ag**L**$_{0.5}$(NO$_3$)] (**2**) and a 1-D polymer [Ag**L**]ClO$_4$ (**3**),, respectively. X-ray structural studies revealed that these polymers are constructed by secondary intermolecular forces such as H-bonding, van der Waals interaction, and π-π stacking. Polymer **2** exhibits photoluminescence at room temperature in the solid state.

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