

New Silver(I)-P4 Coordination Polymers Strongly Adsorb Congo Red to Yield Composites with Enhanced Photocurrent Responses

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Reaction of tetraphosphine (P4) ligand dppbpda with Ag(I) oxylates yielded new coordination polymers (CPs) of formula [(dppbpda)Ag₄(Ox)₄]_n (Ox=Bz (1), Sal (2), LA (3), Bz=benzoate, Sal = salicylate, LA = lactate). The solid-state structures of 1-3consist of one dimensional (1D) chains constructed from tetranuclear μ - η^2 , η^2 - connected Aq₄(Ox)₄ cluster cores and dppbpda bridging ligands. Compounds 1-3 absorbed Congo Red in two distinct steps, with maximum uptake quantities of 1438 (1), 1417 (2) and 1656 (3) mg/g, inversely correlating with the size of the anions in the CPs. These CPs had band gaps in the semiconducting region (Eg) 3.05-3.15 eV and all three showed enhanced two-peak photocurrent responses during the stepwise adsorption of CR, with maximum photocurrent densities of 227 (1), 163 (2) and 386 (3) μ A cm⁻² for composites with optimized dye coverage.

In recent years, coordination polymers (CPs) have been designed with metal nodes and tightly-coordinated bridging ligands to be photoelectric materials^[1-3] and photocatalysts with good stability and energy gap (Eg) values in the semiconductor region.^[4] The photoelectric properties of these CPs can be adjusted by changing the metal centers or bridging ligands. Various types of materials with interesting structures and good performance have been produced by employing ligands containing multidentate coordination sites such as carboxylate, pyridyl, Schiff base and other donors, to assembly transition metal cations including Zn(II), Cd(II), Co(III), Ni(II), Cu(I)/Cu(II) and Mn(II).^[5-11] Coating semiconductive substrates with organic dyes can be an effective method to improve the photoelectric performances of these materials for applications including dye-sensitized solar cells^[12-14] and chemolumines-

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cence sensors.^[15,16] Many CPs absorb organic dyes and the composites exhibit improved resulting photoelectric responses.^[17,18] For example, a one dimensional (1D) CP of Zn(II), 1,2,3-BTA (1,2,3-benzenetricarboxylate) and N,N'-di(3-pyridyl)succinamide showed superior Congo Red (CR) sorption up to 1129 mg/g, and the resulting composite exhibited a remarkably enhanced photocurrent response.^[17] Scientists usually aim to maximize the uptake of dye and thereby to maximize photocurrent, but without specifically designing for this parameter.^[19]

PPh₂ containing bridging ligands with Ag(I) and Au(I) cations have been used for the construction of photoelectroactive materials.^[20] We have previously focused on the design of complexes derived from transition metals and multidentate phosphine or PN hybrid ligands. For example, a tetraphosphine (P4) ligand dpppda (N,N,N',N'-tetrakis (diphenylphosphinomethyl)phenyl-4,4'-diamine) composed of four -PPh₂ groups and p-phenylenediamine spacer was combined with AqNO₃ to yield a CP with formula $[Ag_4(NO_3)_4(dpppda)]_{n'}$ that catalyzed the photo-degradation of nitro-aromatics.^[21] Other CPs of similar composition were found to contain the dpppda \cdot^+ cationic radical and exhibited enhanced photocurrent responses.[22] These results encouraged us to design a P4 ligand dppbpda (N,N,N',N'-tetrakis (diphenylphosphinomethyl)biphenyl-4,4'-diamine) with a longer p-diphenylenediamine spacer and to prepare CPs from the reactions of dppbpda with Ag(I) oxylates. Herein, we report the syntheses and structures of these CPs and their blending with Congo Red (CR) to yield semiconductive composites with enhanced photocurrent responses.

The air-sensitive ligand dppbpda was synthesized in 79% yield by a typical Mannich reaction^[23] of benzidine, formaldehyde and HPPh₂ (molar ratio 1:4:4) in toluene at 110°C for 12 h (Scheme 1). Reactions of dppbpda with AgBz, AgSal and AgLA (molar ratios 1:4, Bz = benzoate, Sal = salicylate, LA = lactate) in CH_2CI_2 and MeCN or MeOH, followed by Et₂O diffusion gave single crystals of the three CPs $[(dppbpda)Ag_4(Ox)_4]_n \cdot m(Sol)$ (1 · CH₂Cl₂ · Et₂O, Ox = Bz, yield 68%, $2 \cdot 2CH_2CI_2$, Ox = Sal, yield 75%, $3 \cdot 2.5MeOH \cdot MeCN$, Ox = LA, yield 55%). The guest solvent molecules in the crystals of the CPs evaporated in air to give solventless 1-3, which were stable to air and moisture and insoluble in water and organic solvents. Their elemental analysis data, IR and NMR spectra were consistent with their molecular structures (see Supporting Information).

Single-crystal X-ray diffraction (SCXRD) of 1.CH₂Cl₂.Et₂O, 2.2CH₂Cl₂ and 3.2.5MeOH.MeCN indicated that they crystal-





Scheme 1. Syntheses of dppbpda and CPs 1-3.

lized in the monoclinic $P2_1/c$ (1, 2) and C2/c (3) space groups. Their frameworks were quite similar (Figure 1 and Figure S1), containing a Ag₄(Ox)₄ cluster core of two O atoms and two eight-membered rings from two Ag atoms and two Ox anions. This core is μ - η^2 , η^2 - ligated by the two –PPh₂ groups from the ends of two neighboring dppbpda molecules, while dppbpda ligands bridge neighboring Ag₄(Ox)₄ cluster cores in a μ - η^2 , η^2 - end-to-end coordination mode, thus forming a 1D Zigzag chain extending along the *b* axis (1, 2), or a wave-like chain extending in the [1,0,1] direction (3). There were no noteworthy intermolecular interactions between these chains.

Adding the fine ground powders of 1–3 (10 mg) into aqueous solutions containing excess CR (0.04 M, 28 mg in 10 mL H₂O) caused a decrease in the intensity of the color of the solution and concomitant coloring of the powders. The mixtures were stirred continuously in the dark for two weeks to ensure saturation. The maximum CR uptake amounts were calculated from the decrease of intensity at 497 nm of the solutions to be 1438 (1), 1417 (2) and 1656 (3) mg/g, respectively. These values are significantly larger than those of Fe_xCo_{3-x}O₄ nanoparticles (128.6mg/g),^[24] ZnO@Ze composite (300 mg/g),^[25] Ce(III) doped UIO-66 nanocrystals (826 mg/g)^[26] or carbon hybridized montmorillonite nanosheets C/MMT



Figure 1. A section of the 1D chain in $1 \cdot CH_2CI_2 \cdot Et_2O$. H atoms and solvent molecules are omitted, and –Ph groups of dppbpda and Bz groups are represented as hexagons for clarity. Atom color codes: Ag, turquoise; P, pink; N, blue; O, red; C, gray.

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(840 mg/g).^[27] Likewise, these CPs absorbed more CR than a CP of Zn(II) ions and 1,2,3-BTA (1129 mg/g).^[17] The adsorbed CR couldn't be removed from the composites by washing with EtOH, and SEM images (Figure 2, Figure S2 and Figure S3) indicated the rough surfaces were obviously smoother.

We further examined the absorption kinetics using aqueous solutions containing 10 mg of CP and the maximum uptake amounts of CR (1: 14.3 mg, BET surface 1.9 m²/g; 2: 14.1 mg, BET surface 4.2 m²/g; **3**: 16.5 mg, BET surface 3.0 m²/g). The change in concentration (Figure 3b, Figure S4b and Figure S5b) clearly showed a two-step absorption process with the kinetics of each step obeying a zero-order rate equation (Table 1). For all three CPs, step-1 was complete within 2.25-3.83 h, much faster than step-2 (>100 h). The rate of step-1 followed the order 3 > 1 > 2, which inversely correlates with the anion sizes LA < Bz < Sal. We hypothesize that smaller anions permit closer interaction of CR with the surface of the CPs. By comparison, the rate constant for step 2 was similar in all three CPs. The change in concentration of Na⁺ in the solutions after absorption was measured by ICP and revealed only trace amounts of this counterion, indicating absorption of the salt, rather than an ion exchange. Evaporation of the solution gave hardly any residue consistent with this proposal. It seems



Figure 2. Photos and SEM images of compound 1 (left) and its CR composite (right).



Figure 3. (a) UV-Vis spectra of the CR solutions (0.687 mmol/L, 30 mL) after treatment with 1 (10 mg) for different periods of time. (b) Time dependent concentrations of the CR solutions calculated from UV-Vis absorbance.

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Table 1. Absorption of CR by 1–3.					
Compound	1	2	3		
Total c _{cR} uptake (mmol/L)	0.687	0.678	0.792		
Max. uptake capacity (mg/g)	1438	1417	1656		
c _{CR} after step 1 (mmol/L)	0.503	0.531	0.561		
Step 1 elapsed time (h)	2.25	3.83	2.44		
Step 1 uptake rate constant $(\times 10^{-2} \text{ mmol} \cdot \text{L}^{-1} \cdot \text{h}^{-1})$	8.6	3.7	10.0		
Step 1 uptake amount (mg/g)	385	307	483		
Step 1 uptake percentage	26.8%	21.7%	29.2 <i>%</i>		
Step 2 uptake rate constant $(\times 10^{-2} \text{ mmol} \cdot \text{L}^{-1} \cdot \text{h}^{-1})$	0.32	0.31	0.31		
Step 2 uptake amount (mg/g)	1053	1109	1173		
Step 2 uptake percentage	73.2%	78.3 %	70.8%		
Step 1 linear fit R ²	0.98891	0.95949	0.97433		
Step 2 linear fit R ²	0.98884	0.97232	0.99438		

reasonable that the Ox anions are tightly coordinated with the Ag ions and unlikely to exchange with the larger CR^- anion.

The band gap energies (Eq) of the CPs were determined by the Kubelka-Munk method (Figure S6) to be 3.15 eV (1), 3.08 eV (2) and 3.05 eV (3), which are in the semiconductivity range. The photocurrent responses of 1-3 and their composites with CR were measured using a three electrode cell in 0.1 M Na₂SO₄ aqueous solution under Xe light. The working electrodes were prepared by coating the powders on indium tin oxide (ITO) glasses, with SCE and Pt wire as reference and auxiliary electrodes, respectively. The cyclic voltammetry (CV) curves for 1-3 were measured under Xe light irradiation and in the dark. All three CPs showed strong reduction peaks for $Ag^{I} \rightarrow Ag^{0}$ at -0.6 V (vs SCE) and weak oxidation peaks for Ag⁰ \rightarrow Ag¹ at 0.38 V (vs SCE, Figure S7), indicating that these CPs may decompose to Ag metal at lower potentials, but are stable at higher potentials up to 1.1 V. Therefore, the bias potential was set at 0.75 V to measure the anodic $H_2O \rightarrow O_2$ photocurrents.

Upon repeated light irradiation with a shutter interval of 20s, CPs 1-3 and their CR composites all showed rapid, steady and repeatable photocurrent responses (Figure 4a, Figure S8a and Figure S9a). The composites showed interesting two-peak responses related to the degree of CR uptake (Figure 4b, Figure 4c, Figure S8b, Figure S8c, Figure S9b and Figure S9c). The two peaks for the composites were 227/171 (1@CR), 140/163 (2@CR), 215/386 (3@CR) µA cm⁻², respectively. The maximum photocurrents were 2.3, 3.3 and 4.5 times larger than those of the pure CPs (97 (1), 49 (2) and 85 (3) μ A cm⁻²). The two peaks all occur with composites prepared from the step-1 CR adsorption, even at different uptake amounts. Composites prepared during step-2 CR absorption exhibited reduced photocurrents that faded with increasing CR absorption. The photocurrents of the dye saturated products (CP@CR-s) were 26 $\mu A\,cm^{-2}$ for 1@CR-s, 37 $\mu A\,cm^{-2}$ for 2@CR-s and 52 $\mu A\,cm^{-2}$ for 3@CR-s, all lower than the corresponding pure CPs. This reduction in activity may be attributed to an outer CR layer blocking the light from impinging on the photoactive centers, and potentially blocking the transport of electrons.

This enhancement of photocurrent at lower dye content encouraged us to explore the interaction of CR with the substrate CPs in the early stage of step-1 adsorption. The



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Figure 4. Photocurrent responses of 1 and its CR composites under Xe light irradiation. (a) Photocurrents during 20s-time on/off light intervals. (b) Photo-ocurrent intensities within 280–300s of the composite formation at different CR uptake amounts and (c) an expansion of (b) in the range of 0–100 mg/g CR uptake.

composites with two photocurrent peaks were designated CP@CR-1 and CP@CR-2. The order of photocurrent was 3@CR-2 > 1@CR-1 > 2@CR-2 inversely correlating with the size of the oxylate ligands LA < Bz < Sal. We propose that CR anion binding in 3@CR-2 is tighter because of the smaller LA anion, resulting in more efficient transfer of photo-generated electrons to the surface of 3, with a commensurately stronger photocurrent response.

Raman spectroscopy was used to probe the interaction of CR and the CPs. The Raman spectrum of CR changed significantly when combined with CP1 in 1@CR-1 and 1@CR-s (Figure 5). Upon coating on 1, the ph-N= stretching frequency of CR^[28] at 1157 cm⁻¹ remained unchanged, while the naphthyl ring C–C stretching^[29] at 1352 cm⁻¹ was blue-shifted to 1370 cm⁻¹, suggesting intermolecular π - π interaction with the



Figure 5. Raman spectra (900–1800 cm⁻¹) of 1, 1@CR-1, 1@CR-s and CR.



CP surface. Likewise, the peak at 1248 cm⁻¹ attributed to the S=O stretching frequency^[30] of 1@CR-1 was blue-shifted from its original value of 1210 cm⁻¹ for pure CR. When the coverage of CR on the composite was increased in 1@CR-s, the relative intensity of this peak was reduced, presumably because the $-SO_3^-$ groups of the outer CR can't interact with the surface of 1 directly. The same trends were observed in the Raman spectra of **2**, **3** and their composites (Figure S10). Therefore, we suppose the two-step adsorption composes the first one which involve the interactions between the CPs' surfaces and the $-SO_3^-$ groups of CR, and the second one that only contain the π - π stacking among CR⁻ anions, as well as the ionic bonding between Na⁺ and CR.

In summary, reactions of dppbpda with AgOx (Ox=Bz, Sal, LA) resulted in three CPs 1-3 with similar 1D chain structures constructed from alternating dppbpda ligands and [Ag₄(Ox)₄] cluster cores with μ - η^2 , η^2 - coordination. CPs **1–3** strongly absorbed CR in water with maximum uptake capacities of 1438 (1), 1417 (2) and 1656 (3) mg/g, inversely correlating with the size of the CP anions. These surface absorptions occurred in two steps, a rapid step (2.25-3.83 h, 21.7-29.2% of maximum uptake capacity) and another much slower step. Composites of 1-3 with CR showed enhanced photocurrent responses with maximum current densities of 227 (1), 163 (2), 386 (3) uA cm⁻², which are 2.3, 3.3 and 4.5 times larger than those of pure 1-3. These photocurrents exhibited two peak responses related to the packing of CR on the surface of the CPs. These observations are useful for the rational design of more efficient photoelectric composite materials and we are currently pursuing this investigation.

Deposition Number(s) 2071252 (for $1 \cdot CH_2CI_2 \cdot Et_2O$), 2071253 (for $2 \cdot 2CH_2CI_2$), and 2071254 (for $3 \cdot 2.5MeOH \cdot MeCN$) contain the supplementary crystallographic data for this paper. These data are provided free of charge by the joint Cambridge Crystallographic Data Centre and Fachinformationszentrum Karlsruhe Access Structures service www.ccdc.cam.ac.uk/structures.

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Conflict of Interest

The authors declare no conflict of interest.

Keywords: Adsorption · Congo Red · Coordination polymers · Photocurrent response · Silver · Tetraphosphine ligands

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