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### Preparation of silver nanoparticles from silver(I) nano-coordination polymer

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### 1. Introduction

Silver has been extensively used in a variety of applications such as catalysis, electronics, photonics and photography due to its unique properties. For example, silver has the highest electrical conductivity, thermal conductivity and reflectivity of all metals [1]. Several methods can be applied to synthesize silver nanoparticles with well-defined shapes. The majority of the more straightforward approaches are based on the reduction of silver nitrate by sodium borohydride [2] or sodium citrate [3]. Some of the more diverse methods previously used include microwave plasma synthesis [4], electrolysis of Ag salts [5], rapid expansion of supercritical solvents [6,7], microemulsion [8], and photoreduction of Ag ions [9,10]. Recently in the last few years several methods for synthesis of silver nano structures have been developed too, which are: (a) synthesis of fine silver powder with a particle size range of 50-1000 nm in a mechanochemical process by inducing a solid-state displacement reaction between AgCl and sodium [11], (b) synthesis of long silver nanowires with lengths of more than 50 mm, some even more than 100 mm, and average diameters of about 80 nm at room temperature by a simple and fast process derived from the development of photographic films [12], (c) synthesis of metallic silver by direct hydrogen reduction of Ag<sub>2</sub>S in basic slurry under hydrothermal conditions [1] and (d) in situ preparation of Ag nanoparticles from the hydrolytic decomposition of silver triethanolamine (TEA) complexes that lead to the formation of spherical metallic silver particles with mean diameter of 8 nm well

### ABSTRACT

Nanorods of two-dimensional organometallic coordination polymer,  $[Ag(\mu_4-DPOAc)]_n$  (1)  $[DPOAc^- =$  diphenylacetate], has been synthesized by the reaction of potassium diphenylacetate (DPOAcK) and AgNO<sub>3</sub> by sonochemical process. Reaction conditions, such as the concentration of the initial reagents played important roles in the size and growth process of the final product. Silver nanoparticles were synthesized from nanorods of compound **1**. These nano-coordination polymer and nanoparticles were characterized by X-ray powder diffraction (XRD) and scanning electron microscopy (SEM). Thermal stability of nano and crystal samples of compound **1** were studied and compared with each other.

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adsorbed onto the bacterial cellulose fibrils [13]. In recent years silver(I) complexes have found application as precursors for Chemical Vapor Deposition (CVD) [14–17]. As mentioned above preparation of silver nanoparticles from silver complexes reported by Barud et al. [13], but the use of silver coordination polymers (CPs) or silver nano-coordination polymers (NCPs) to prepare silver nano structures is sparse [18]. NCPs are generally synthesized by exploiting the insolubility of the particles in a given solvent system. Wang et al. reported the first synthesis of NCP in 2005 [19]. They isolated monodisperse spheres of approximately 420 nm in diameter, taking advantage of the very low solubility in water of the product from the reaction between the initial precursors. We used this method to prepare NCPs by sonochemical process [20-25]. Continuing our previous work on CPs [26-28,25,29-31], synthesis of Ag<sup>I</sup> NCP by sonochemical process and use this precursor to fabricate silver nanoparticles with oleic acid as a surfactant were reported.

### 2. Experimental

### 2.1. Materials and physical techniques

All reagents for the synthesis and analysis were commercially available and used as received. Double distilled water was used to prepare aqueous solutions. A multiwave ultrasonic generator (Sonicator\_3000; Misonix, Inc., Farmingdale, NY, USA), equipped with a converter/transducer and titanium oscillator (horn), 12.5 mm in diameter, operating at 20 kHz with a maximum power output of 600 W, was used for the ultrasonic irradiation. Microanalyses were carried out using a Heraeus CHN–O– Rapid analyzer.



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Melting points were measured on an Electrothermal 9100 apparatus and are uncorrected. IR spectra were recorded using Perkin–Elmer 597 and Nicolet 510P spectrophotometers. The thermal behavior was measured with a PL-STA 1500 apparatus between 20 and 780 °C in a static atmosphere of nitrogen. Crystallographic measurements were made using a Bruker APEX area-detector diffractometer. The intensity data were collected using graphite







**Fig. 1.** (a) Molecular structure representations of **1**, showing Ag—Ag and Ag–C distances and Ag atom environment, (b) a fragment of the two-dimensional network in **1**, (c) space filling representation of **1**. (Ag = violet, O = red, C = gray). (For interpretation of the references to color in this figure legend, the reader is referred to the web version of this article.)

monochromated Mo K $\alpha$  radiation. The structure was solved by direct methods and refined by full-matrix least-squares techniques on  $F^2$ . Structure solution and refinement was accomplished using SHELXL-97 program packages [32]. The molecular structure plot and simulated XRD powder pattern based on single crystal data were prepared using Mercury software [33]. X-ray powder diffraction (XRD) measurements were performed using a Philips X'pert diffractometer with mono chromatized Cu k $\alpha$  radiation. The samples were characterized with a scanning electron microscope with gold coating.

## 2.2. Synthesis of $[Ag(\mu_4-DPOAc)]_n$ (1) and preparation of its single crystals

In 20 ml CH<sub>3</sub>CN 0.212 g (1 mmol) 2,2-diphenylacetic acid were mixed and stirred with a solution of 0.057 g (1 mmol) KOH in 3 ml H<sub>2</sub>O to form a clear solution. Addition of 0.170 g (1 mmol) AgNO<sub>3</sub> in 5 ml H<sub>2</sub>O produced a clear solution. The resulting solution was stirred and then allowed to stand in darkness at room temperature



**Fig. 2.** XRD patterns; (a) simulated pattern based on single crystal data of compound **1**, (b) nanorods of compound **1** prepared by sonochemical process, (c) spongy silver prepared by calcinations of compound **1** at 873 K, (d) silver nanoparticles prepared by OA at 453 K.

to evaporate for several days to obtain suitable crystals. The crystals were washed with acetone and air dried, d.p. = 235 °C, Yield: 0.112 g (35% based on final product). IR (selected bands; in cm<sup>-1</sup>): 554m, 637m, 691s, 723s, 752m, 1023w, 1068w, 1267w, 1328w, 1374vs, 1437w, 1479m, 1531vs and 1589m. *Anal.* Calc. for  $C_{14}H_{11}AgO_2$ : C, 52.64; H, 3.45. Found: C, 52.50; H, 3.34%.

# 2.3. Synthesis of $[Ag(\mu_4-DPOAc)]_n$ (1) nanopowders by sonochemical process

To prepare the nanorods of  $[Ag(\mu_4-DPOAc)]_n$  by sonochemical method, a high-density ultrasonic probe immersed directly into the solution of DPOAcK (50 ml, 0.05 M) in double distilled water, then into this solution, a proper volume of AgNO<sub>3</sub> aqueous solution (50 ml, 0.05 M) was added in drop wise manner. The solution was ultrasonically irradiated with the power of 12 W for 1 h. The obtained precipitates were filtered, subsequently washed with double distilled water and then dried, d.p. = 200 °C, (Found C: 52.40, H: 3.37%). IR (selected bands; in cm<sup>-1</sup>): 555m, 635m, 690s, 727s, 755m, 1027w, 1069w, 1268w, 1327w, 1371vs, 1434w, 1479m, 1530vs and 1589m. Certainly we should mention that different concentrations of metal and ligand solutions (0.0125, 0.025 and 0.125 M) with the power of 12 W for 1 h were tested but appropriate nanorods of compound 1 with the best morphology and dispersion obtained under the mentioned conditions. In order to obtain silver nanoparticles, precipitate of compound **1** nanorods which obtained from 0.05 M solutions of initial reagents was calcinated at 873 K in a furnace and static atmosphere of air for 5 h.

### 2.4. Synthesis of silver nanoparticles by surfactant

To prepare silver nanoparticlas by surfactant [34], precipitates of compound **1** nanorods (160 mg, 0.5 mmol) was dissolved in oleic acid (OA), (8 mL, 25 mmol) and formed a yellow solution. This

solution was degassed for 15 min and then heated to 180 °C for 1 h under air atmosphere. At the end of the reaction, a black precipitate was formed. A small amount of toluene and a large excess of EtOH were added to the reaction solution, and metallic silver was separated by centrifugation. The solid was washed with EtOH and dried, neither d.p. and nor IR bands was observed.

### 3. Results and discussion

The reaction between diphenylacetate (DPOAc<sup>-</sup>) and Ag(NO<sub>3</sub>) provided a crystalline material of the general formula  $[Ag(\mu_4-$ DPOAc)]<sub>n</sub> (1) [27]. The structure determination of 1 by X-ray crystallography showed that the silver atoms can be considered to be three-coordinate with AgO<sub>3</sub> coordination sphere and Ag-O distance of 2.2063(17), 2.2519(17) and 2.4872(18) Å. Ag-O bonds in similar compounds have distances in the range of 2.21-2.64 Å [35–40]. The carboxylate group of the DPOAc<sup>-</sup> ligand in compound 1 acts as a bridging group where each oxygen atom of the carboxvlate group coordinates to a silver(I) ion, and one of these oxygen atoms also bridges to one other silver atom (Fig. 1a). A search was made generally for Ag...Ag approaches and it appears that argentophilic interactions could be observed in compound 1 with distance of 2.8463(4) Å between two Ag1 atoms. This Ag-Ag interaction in compound **1** are longer than the Ag–Ag distances in similar dinuclear complexes (i.e., 2.704(2), 2.669(1), and 2.726(1)Å) [41–46] and slightly shorter than those in the polymeric structure [47–50]. The relatively short Ag–Ag bonds found here may thus be considered to be only d<sup>10</sup>...d<sup>10</sup> noncovalent interactions [44-50]. A general search was also made for Ag-C approaches in compound 1 and it appears that the Ag atoms may unexpectedly be involved in an  $\eta^1$  interaction with the C<sub>phenvl</sub> atom of neighboring molecules with a Ag(1)-C(5) distance of 2.710(3) Å. In compounds with similar bonds the Ag-C distances are in the range of 2.40–2.70 Å [51–54], in [Ag(benzene)ClO<sub>4</sub>] for example



Fig. 3. SEM images of compound 1 nanorods obtained in various concentration of initial precursor; [Ag<sup>+</sup>] = [DPOAc<sup>-</sup>] = (a) 0.125 M, (b) 0.05 M, (c) 0.025 M and (d) 0.0125 M.

they are 2.496 and 2.634 Å [45]. Some other Ag(I) polymeric complexes with Ag–C (sp<sup>2</sup>) bonded polycyclic aromatic ligands have been reported to have mean Ag–arene distances of 2.82–3.37 Å [55–62]. Thus compound **1** can be considered to contain silver atoms with fourfold coordination and an O<sub>3</sub>CAg...AgO<sub>3</sub>C environment (Fig. 1a). Thus, strong monohapto aromatic coordination of Ag atoms in the compound **1** could be considered and appears to be yet another factor which can make varying contributions to the construction of an organometallic coordination polymer. The structure of compound **1** may also be considered as a coordination polymer of Ag(I) consisting of one-dimensional linear chains constructed of carboxylate group bridging, running parallel to the *b* axis and the individual polymeric chains are almost parallel to each other and further bridged by Ag–C bonds, resulting in a twodimensional framework as shown in Fig. 1b.

Fig. 2a shows the simulated XRD pattern from single crystal Xray data of the above compound and Fig. 2b shows the XRD pattern of a typical samples of  $[Ag(\mu_4-DPOAc)]_n$  (1) prepared by the sonochemical process. Acceptable matches, with slight different in  $2\theta$ , were observed between the simulated from single crystal X-ray data patterns (Fig. 2a) and those from the experimental powder X-ray diffraction patterns for nanopowders crystalline sample as obtained from the synthesis by sonochemical process (Fig. 2b). Results of XRD powder patterns indicate that the experimental data are in good agreement with the simulated XRD powder patterns based on single crystal data, hence this compound obtained as a mono-phase. The morphology, structure and size of the four samples which were prepared with different concentrations of AgNO<sub>3</sub> and DPOAcK are investigated by Scanning Electron Microscopy (SEM). Fig. 3a-d indicates that the original morphology of the nanorods obtained by sonochemical process. As could be observed



Fig. 4. Thermal behavior of compound 1 (a) single crystals and (b) nanorods.

from this figure, concentration increase from 0.0125 M (Fig. 3d) to 0.05 M (Fig. 3b) results in monotonousness and non tenacity of the nanorods, but the results of concentration increase from 0.05 M (Fig. 3b) to 0.125 M (Fig. 3a) was not satisfactory. The best result attributed to 0.05 M solution of Ag<sup>+</sup> ion and DPOAc<sup>-</sup> ligand that shows the formation of silver nanorods with the diameter of about 80-250 nm (average diameter of 150 nm) and the length of about  $0.5-3.0 \ \mu$ m (average length of 2  $\mu$ m) as shown in Fig. 3b.

Thermal gravimetric (TG) and differential thermal analyses (DTA) of compound **1** single crystals shows that the crystalline form is very stable and does not decompose up to  $235 \,^{\circ}$ C (Fig. 4a), at which temperature pyrolyze of compound **1** starts. In



**Fig. 5.** SEM images of silver spongy nano structure prepared by calcination of compound **1** at 873 K (up), nanorods of compound **1** prepared by sonochemical process (middle), silver nanoparticles prepared by OA at 453 K (down).

this stage, exothermic removal of DPOAc<sup>-</sup> occurs between 235 and 480 °C with a mass loss of 63.0% (Calc. 63.6%). Mass loss calculations and XRD pattern (Fig. 2c), shows that the final decomposition product is metallic silver. Nanorods of compound **1** are much less stable at starts to decompose at 200 °C (Fig. 4b). The TG curve exhibits a distinct decomposition stage between 200 and 430 °C with a mass loss of 64.2% (calcd 63.6%). Decomposition of compound **1** nanorods starts at about 35° earlier than its single crystals, probably due to different contact between crystals and the pan, size, shape and compactness of the two samples or due to more heat that needed to annihilate the lattices of single crystal. This stabilization is annihilated approximately by produce nanorods of this compound by sonochemical process.

Fig. 2c shows the XRD patterns of the residue obtained from calcinations of compound **1** nanorods at 873 K. The obtained patterns match with the standard patterns of cubic silver with the lattice parameters (a = 4.0862 Å and z = 4) which are close to the reported values, (JCPDS card number 04-0783), but SEM image shows that agglomeration was occurred in this process (Fig. 5 up) and a spongy solid, due to pyrolyze of DPOAc<sup>-</sup> and remove of the resulting gas, was formed. In order to obtain silver nano structures from NCP of **1** and to prevent from agglomeration, we use oleic acid as a surfactant to thermal decomposition of compound 1 occurs in the resulting micelles [34]. XRD patterns of residue (Fig. 2d), shows that the resulting residue was cubic silver with the lattice parameters mentioned above. SEM image of the resulting residue shows the formation of silver nanoparticles with the average diameter of about 95 nm (Fig. 5 down). In our previous work on silver(I) NCP, we observed that silver nanoparticles obtained from 3D coordination polymer with no argentophilic interactions in the resulting network [18]. In this work, we obtained similar results from 2D coordination polymer with no argentophilic interactions as a chain or grid in the compound 1 network. As could be seen from Fig. 1c, silver atoms are separated from each other and surrounded with aromatic phenyl rings. These aromatic phenyl rings and the resulting structure could be considered as a template to fabricate silver nanoparticles [63]. Probably we could conclude that silver nanoparticles are obtained from 2D or 3D coordination polymer with no argentophilic interactions as a chain, grid or network in the resulting structure.

### 4. Conclusions

Nanorods of an organometallic coordination polymer of silver(I) with a less-common  $\eta^1$ -coordination mode of the phenyl rings was synthesized by sonochemical process. Concentration increase of the initial reagents results in formation of compound **1** nanorods with the best morphology. Decreasing the size of this coordination polymer to nanometer results in less thermal stability of this compound compared with its single crystal sample. Calcinations of compound **1** nanorods to fabricate silver nano structures was not successful. In order to obtain silver nanoparticles from compound **1**, oleic acid was used as a surfactant to thermal decomposition of compound **1** and formation of silver nanoparticles occurs in the resulting micells at 180 °C. We could conclude that silver nanoparticles are obtained from 2D or 3D coordination polymer with no argentophilic interactions as a chain, grid or network in the resulting structure.

#### 5. Supplementary material

CCDC 639612 contains the supplementary crystallographic data for this paper. These data can be obtained free of charge from The Cambridge Crystallographic Data Center via www.ccdc.cam.ac.uk/ data\_request/cif.

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