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Synthesis and Characterization of Silver Choromate Nanostructures via a Simple Precipitation Method

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In this work, Ag_2CrO_4 nanostructures have been synthesized via a precipitation method using silver salicylate, [Ag(HSal)], as a new precursor. At first, silver salicylate was prepared at room temperature through a precipitation method. So, silver nitrate and sodium salicylate were used as starting materials. Besides, the effect of silver precursor and surfactant concentration on the morphology of the products was investigated by SEM images. SEM images showed that particle-like powders with particle size of 250–300 nm and capsule-like nanostructures of Ag_2CrO_4 with diameters ~ 100 nm and lengths 130–140 nm have been produced using $AgNO_3$ and [Ag(HSal)], respectively. In several experiments to decrease the particle size of products, sodium dodecyl sulfate (SDS) was applied as surfactant. The as-synthesized products were characterized by energy dispersive spectrometry (EDS), powder X-ray diffraction (XRD), thermogravimetric and differential thermal analyses (TGA/DTA), scanning electron microscopy (SEM) and FT-IR (Fourier transform infrared spectroscopy).

Keywords: Silver Chromate, Precursor, Precipitation Method, Nanostructures, Surfactant.

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1. INTRODUCTION

Recently, the shape and size of inorganic nanomaterials are well known to have an important influence on their widely varying electrical and optical properties [1], which are important in various applications. Among these nanomaterials, it was found that silver chromates are a good visible-light sensitive photocatalyst [2].

Ag_2CrO_4 can be used as cathode for lithium cells [3], solid electrolyte system involving CuI and Ag_2CrO_4, and ion transport, electrical and electrochemical properties [4]. Silver iodide solid electrolytes, containing dichromate anion (AgI-$Ag_2Cr_2O_7$) behave as supercooled liquids [5].

So far, there are a few reports on the synthesis of Ag_2CrO_4 nanostructures. Liu et al. prepared necklace structures of Ag_2CrO_4 composed of single crystalline nanorods with diameters of about 40 nm and lengths of 300 nm using high-active acrylicamide template [6]. Alamdari and co-workers synthesized Ag_2CrO_4 nanoparticles by precipitation method using $AgNO_3$ and K_2CrO_4 as starting reagents, and they studied the effect of silver and chromate concentrations, flow rate of reagent addition and temperature on the particle size of synthesized silver chromate particles [7]. In addition, biomimetic synthesis of Ag_2CrO_4 quasi-nanorods and nanowires by emulsion liquid membranes was reported by Liu et al. [8]. On the other hand, a major interest is in the development of organometallic or inorganic compounds as precursor for the preparation of nano-sized materials [9, 10]. The aim of this study was to investigate the effect of some experimental parameters on the morphology of Ag_2CrO_4 nanostructures and to find the best experimental conditions for the synthesis of Ag_2CrO_4 nanostructures via a precipitation method.

2. SYNTHESIS AND CHARACTERIZATION

2.1 Method of Sample Manufacturing and Analysis

Silver(I) salicylate, [Ag(HSal)], was synthesized according to this procedure: 4 mmol of $AgNO_3$ was dissolved in 50 mL of distilled water. A stoichiometric amount of sodium salicylate dissolved in an equal volume of distilled water was added drop-wise to the above solution under magnetic stirring. After stirring for 15 min at room temperature, a white precipitate was obtained, isolated and washed with distilled water and ethanol several times to remove impurities. The as-synthesized white precipitate was dried at 50 °C in vacuum, and characterized by ^1H-NMR, and TGA/DTA.

In a general procedure, Ag_2CrO_4 was prepared by reaction between silver precursor and Na_2CrO_4 with molar ratio of 2:1. At first, 0.002 mol of silver salicylate was dissolved in 50 mL of distilled water and then, a solution including 0.001 mol of Na_2CrO_4 dissolved in 50 mL of distilled water was added into the above solution drop-wise for 10 min under vigorous magnetic stirring. After the addition of the Na_2CrO_4, the solution was stirred for 10 min, and the obtained precipitate collected and washed repeatedly with distilled water and ethanol several times and finally dried at 50 °C in vacuum. The as-synthesized Ag_2CrO_4 nanostructures were characterized by SEM, FT-IR, EDS, and XRD analyses.

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3. RESULTS AND DISCUSSION

Fig. 1a shows the TGA/DTA of [Ag(HSal)] precursor. As shown in Fig. 1a, an exothermic stage of [Ag(HSal)] decomposition occurs between 210 and 310 °C with a mass loss of 56.3 % (calcd 55.80 %). Mass loss calculations showed that the final decomposition products were AgO and Ag_2O. Fig. 1b shows the ^{1}H-NMR spectrum of precursor. The multiple peaks appeared at the aromatic protons. The sharp peak appeared at

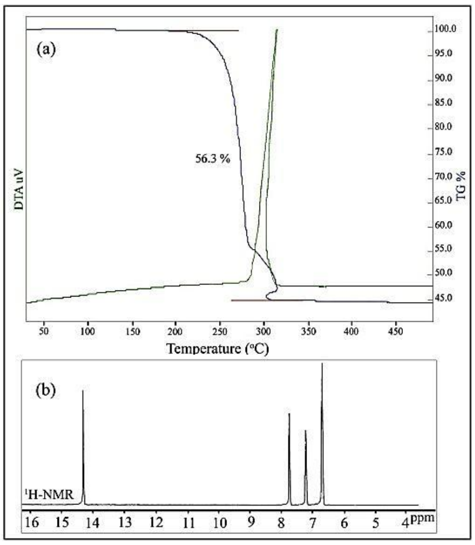

Fig. 1 – TGA/DTA curves (a), and 1H-NMR spectrum (b) of [Ag(HSal)]

chemical shifts of 6.6–7.8 ppm could be assigned to 14.35 ppm could be assigned to the proton of phenolic hydroxyl group. The chemical shift of hydroxyl group showed that this group could interact with silver ion.

In the EDS spectrum of sample 4 (Fig. 2a), Ag, Cr, and S elements are detected. The presence of sulfur element in this spectrum is because of Ag_2CrO_4 capped by SDS. (Fig. 2b), shows FT-IR spectrum of sample 4 in the range 400–4000 cm^{-1}. The FT-IR spectrum of Ag_2CrO_4 obtained from sample 4 shows a strong absorption band at 888.45 cm^{-1}, which may be assigned to the υ_3 vibration modes of Cr–O [11].

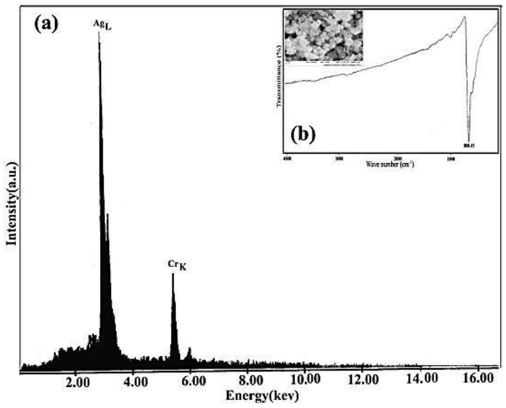

Fig. 2 – EDS spectrum (a), and FT-IR (b) of Ag_2CrO_4

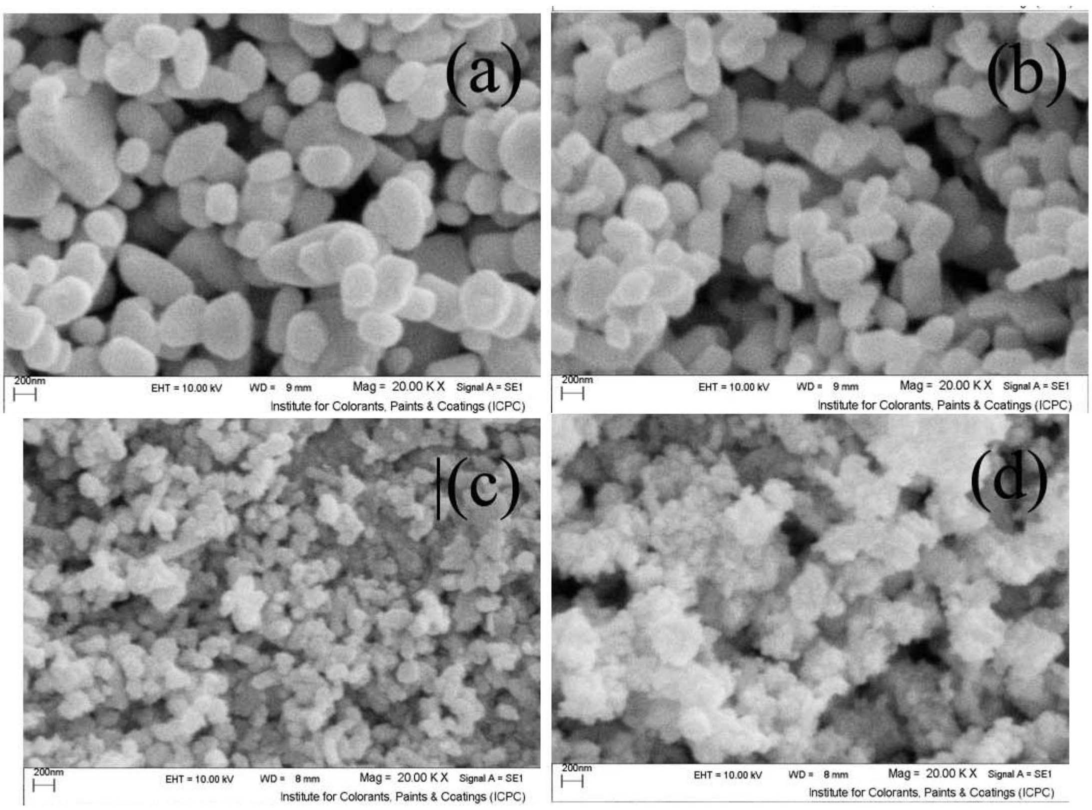

Fig. 4 – SEM images of samples 1 (a), 2 (b), 3 (c), 4 (d)

Fig. 3 shows the XRD pattern of Ag_2CrO_4 obtained from sample 4. All of the reflection peaks in Fig. 3 can be readily indexed to a pure orthorhombic phase of Ag_2CrO_4 (JCPDS No. 26–0952).

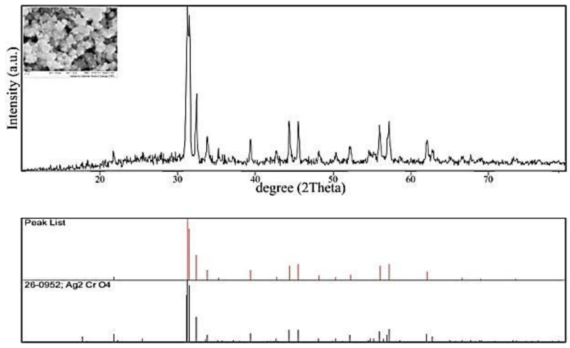

Fig. 3 – XRD pattern of Ag_2CrO_4

SEM image of sample 1 is shown in Fig. 4a. When $AgNO_3$ was used as silver precursor, particle-like microstructures of Ag_2CrO_4 with particle size of 250–300 nm were obtained. To modify the morphology of products, silver salicylate was used as silver source.

By using [Ag(HSal)] as precursor (sample 2), capsule-like nanostructures of Ag_2CrO_4 with diameter of about 100 nm and length of 130–140 nm were obtained (Fig. 4b). To decrease the particle size of Ag_2CrO_4, SDS was applied. When the amount of SDS was 0.1 g (Fig. 4c) and 0.5 g (Fig. 4d), particle-like nanostructures of Ag_2CrO_4 with particle size of 80–90 and 30–35 nm were produced respectively.

CONCLUSIONS

In summary, Ag_2CrO_4 nanostructures were prepared via a simple precipitation method by using [Ag(HSal)] as a new silver precursor. According to SEM images, the morphology of silver chromate nanostructures was 1-D and 3-D by using [Ag(HSal)] and $AgNO_3$, respectively. Besides, SDS molecules were applied to decrease the particle size of the as-synthesized products.

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REFERENCES

1. C. M. Lieber, *Solid State Commun.* **107**, 607 (1998).
2. S. Ouyang, Z. Li, Z. Ouyang, T. Yu, J. Ye, Z. Zou, *J. Phys. Chem. C* **112**, 3134 (2008).
3. G. Eichinger, J. O. Besenhard, *J. Electroanal. Chem. Interface. Electrochem.* **72**, 1 (1976).
4. S. A. Suthanthiraraj, Y. D. Premchand, *J. Solid State Chem.* **177**, 4126 (2004).
5. M. Lazzari, B. Scrosati, C. A. Vincent, *Electrochimica. Acta* **22**, 51 (1977).
6. J.-K. Liu, C.-X. Luo, N.-J. Quan, *J. Nanopart. Res.* **10**, 531 (2008).
7. R. F. Alamdari, S. S. Hajimirsadeghi, I. Kohsari, *Inorg. Mater.* **46**, 60 (2010).
8. L. Liu, Q. Wu, Y. Ding, H. Liu, J. Qi, Q. Liu, *Aust. J. Chem.* **57**, 219 (2004).
9. F. Mohandes, F. Davar, M. Salavati-Niasari, *J. Magn. Magn. Mater.* **322**, 872 (2010).
10. F. Mohandes, F Davar, M. Salavati-Niasari, K. Saberian, *Current Nanoscience* **7**, 260 (2011).
11. M. Cieglak-Golonka, *J. Thermal. Anal.* **38**, 2513 (1992).