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A modified Solvothermal approach for developing Au/SnO₂ nanocomposites

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ABSTRACT

We report a modified solvothermal route to synthesize Au/SnO₂ nanocomposites. A rapid one step wet chemical procedure is adopted to synthesize colloidal gold nanoparticles, by mixing an aqueous solution of HAuCl₄, CTAB and NaBH₄ in appropriate ratio. The gold solution is added to the mixture containing SnCl₂, NaOH and CTAB in an appropriate ratio and solvothermal treatment is adopted for the formation of Au/SnO₂ nanocomposites. The as-formed Au/SnO₂ nanocomposite was subjected to structural, optical and morphological characterization studies.

Keywords: gold nanocomposites, nanocomposites, Au/SnO₂, solvothermal.

INTRODUCTION

Recently, materials with multicomponent functional properties have been the subject of extensive research in materials science. The functional properties of semiconductor and metal nanoparticles (NPs), such as catalytic or sensing properties or tunable luminescence, can be greatly enhanced by the formation of nanocomposites. Nanocomposites also offer the possibility to combine diverse properties which are impossible within a single material [1]. Tin dioxide is an n-type semiconductor oxide with a wide energy gap. Tin dioxide (SnO₂) has attracted much attention for its various applications, such as catalyst, catalytic support, biomedical, pharmaceutical, gas sensor, rechargeable Li battery and optical electronic device [2-4]. But the catalytic activity of gold supported on tin dioxide is gearing up attention in recent years. It is observed that these nanocomposites showed excellent catalytic property of CO oxidation at low temperatures. Hence it is quite needed to focus on the more effective synthetic procedure for developing Au/SnO₂ nanocomposites. A scan on literature indicates that a variety of methods for nanocomposite synthesis have been tested, including sol-gel, laser ablation, spray pyrolysis, sputtering and successive ionic layer deposition (SILD) method [5-9]. But synthesis through solvothermal method is rarely reported. Solvothermal synthesis is yet another simple and effective method to synthesize nanocomposites. The solvothermal method is one of the best wet chemical methods for the preparation of high quality oxide powder and the powder prepared by hydrothermal process has advantages such as well-defined grain, no aggregation and good dispersivity [10]. Hence in this work we have made an attempt to synthesize Au/SnO₂ nanocomposites via a modified solvothermal route.

MATERIALS AND METHODS

2.1. Materials and synthesis

Hydrogen tetrachloroaurate(I) hydrate ($\text{HAuCl}_4 \cdot \text{H}_2\text{O}$, 99.9+%) (Loba Chemi), trimethyl ammonium bromide (CTAB, 98%) (Loba Chemi), sodium borohydride, $\text{SnCl}_2 \cdot 2\text{H}_2\text{O}$ (Merck), NaOH (Fisher scientific). All chemicals were used as received. Ultra pure water from Millipore was used to prepare all solutions.

2.2. Synthesis of gold nanoparticles (solution A)

Initially, 50 ml of 0.747×10^{-3} M of $\text{HAuCl}_4 \cdot \text{H}_2\text{O}$ aqueous solution was taken and 2.025×10^{-2} mol of CTAB powder was added to the aqueous solution of HAuCl_4 . With this addition, the solution turned light orangish yellow colour. Then 750 μl of 0.15 M of NaBH_4 is added to the above mixture. The colour of the solution turned wine red indicating the formation of gold nanoparticles. The pH was found to be 7. The solution was labeled as A.

2.3. Synthesis of SnO_2 nanoparticles (solution B)

5ml of 0.3M $\text{SnCl}_2 \cdot 2\text{H}_2\text{O}$ was taken and then 50 ml of 0.3M NaOH was added to it and stirred well, till a clear solution was seen. 2 m mol of CTAB was added to the above mixture. The final mixture was stirred till the CTAB got completely dissolved in the solution. The pH was found to be 14. The solution was labeled as B.

2.4. Synthesis of Au/ SnO_2 nanocomposites

The solution A was poured into solution B and stirred for a few minutes. The solution mixture turned pinkish white colour. The above mixture was transferred to an Teflon-lined autoclave and kept for 180°C for 24 h. The resultant mixture was centrifuged, washed and the final product was dried at 70°C for 5 h and then collected.

2.4. Characterization techniques

X-ray diffraction analysis of the as-prepared sample was done using RICH SEIFER with monochromatic nickel filtered CuK_α ($\lambda=1.5461 \text{ \AA}$) radiation with the scanning rate of 0.02° . For transmission electron microscopic (TEM) analysis, the sample was dispersed in ethanol and ultrasonicated for 30 min. The analysis was carried out for different magnifications by JEOL JEM 3010 operating at 200 kV. Scanning electron microscope (SEM) was employed for morphological study of the sample using a JEOL JSM 6310 operated at 15 kV. The UV-Visible spectrum of the sample was recorded in the spectral range of 200–800 nm using a UV-visible spectrophotometer (UV-2450, Shimadzu).

RESULTS AND DISCUSSION

3.1. Powder XRD analysis

Fig. 1 shows the XRD patterns of the as prepared Au/ SnO_2 nanocomposites. All the main peaks in this pattern are strong and can be indexed to the tetragonal rutile SnO_2 structure (JCPDS 21-1250). But the diffraction peaks are broadened due to the small size of the particles. In addition to the peaks of SnO_2 , three weak diffraction peaks, attributing to Au (1 1 1), Au (2 0 0) and Au (2 2 0) planes, are also observed (JSPDS card No.04-0784). The appearance of these peaks confirms that crystalline Au and SnO_2 phases are formed in Au/ SnO_2 nanocomposites, No peaks belonging to other chemicals are found. This confirms that the as prepared sample is in the pure form.

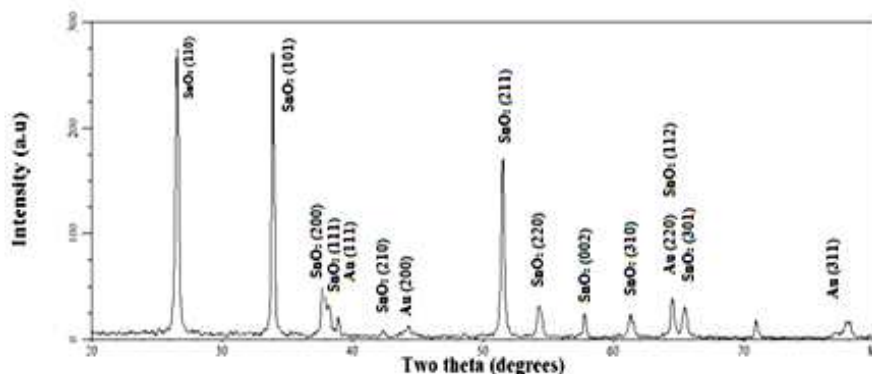


Fig.1 Powder X-ray Diffraction of Au/ SnO_2 nanocomposite

3.2. UV-Vis absorption study

The UV-Vis spectrum is useful for the understanding of the structural variation of materials via calculating the bandgap values. Fig. 2 shows the UV-Vis absorption spectrum of Au/SnO₂ nano composites. The spectrum shows two broad peaks one centered around 263 nm due to the presence of SnO₂, and the other peak around 543 nm is due to the surface Plasmon resonance of the gold nanoparticles formed. This confirms the presence of Au/SnO₂ nanocomposites. Further the observed red shift of around 24 nm when compared to pure gold (519 nm) indicates the formation of Au/SnO₂ nanocomposite [11].

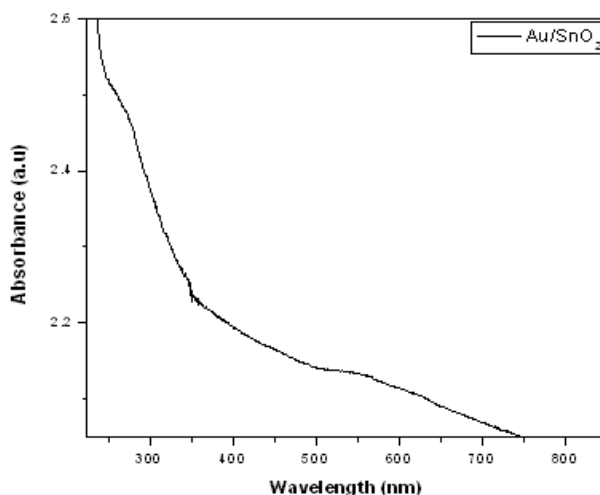


Fig.2 UV-Vis absorption spectrum of Au/SnO₂ nanocomposite

3.3. SEM analysis

Fig. 3a shows the SEM image of the as-prepared Au/SnO₂ nanocomposites. The SnO₂ particles are on the shape of nanoplates. The SnO₂ particles are clear and have a sharp morphology. The gold nanoparticles are found deposited on the nanoplates of SnO₂. Fig.3b shows the EDX spectrum of the Au/SnO₂ nanocomposites. The spectrum shows the presence of Sn, O₂ and Au. This confirms formation of Au/SnO₂ nanocomposites and there is no trace of any kind of impurity.

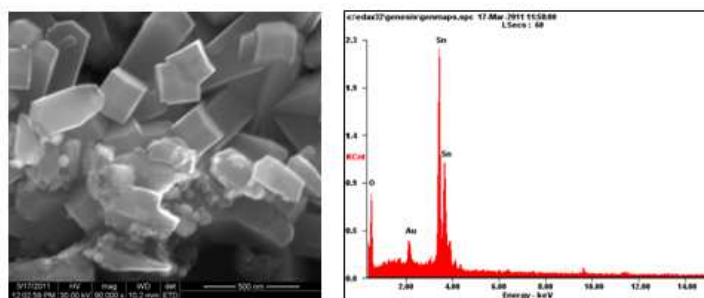


Fig.3 a. SEM image of Au/SnO₂ nanocomposite and b. EDX spectrum of Au/SnO₂ nanocomposite

3.4. TEM analysis

Fig.4 a shows the transmission electron microscopic image of Au/SnO₂ nanocomposites. The image clearly shows the presence of SnO₂ as nanoplates with decorated gold nanoparticles. The gold nanoparticles clearly seen embedded on the nanoplates of SnO₂. The size of the gold nanoparticles are around 20nm. Fig.4b shows the HRTEM image of the Au/SnO₂ nanocomposites. The lattice fringes of the gold and SnO₂ nanoparticles are clear. The HRTEM image of the Au/TiO₂ nanocomposite reveals that the well-defined interface and continuity between metallic Au nanoparticles and SnO₂ layer is formed which is indicative of the strong interaction between the exposed Au atoms and SnO₂ bonds in SnO₂ crystals. The observed result fairly matches with the reported work of Yang Tian-Zu et al [11] who have developed nanoplates of SnO₂ embedded with Au nanoparticles.

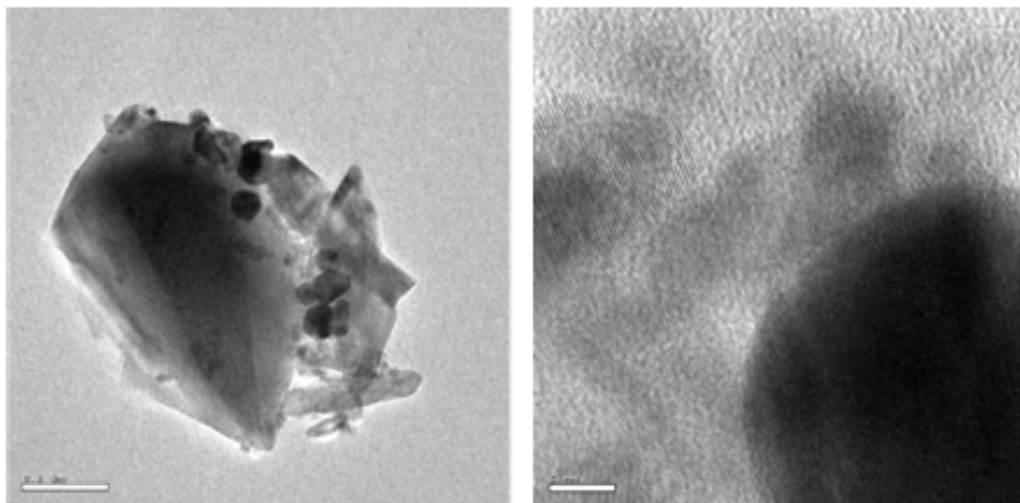


Fig.4a. TEM image of Au/SnO₂ nanocomposite b. HRTEM image of the Au/SnO₂ nanocomposite

CONCLUSION

We have demonstrated a simple and flexible solvothermal route for the synthesis of Au/SnO₂ nanocomposites. The nature of the particles is studied under both microscopic and spectroscopic characterization. The UV-Vis absorption spectrum of the Au/SnO₂ nanocomposites shows the presence of SnO₂ and gold nanoparticles in the composite. The EDX spectrum shows the presence of both SnO₂ and gold. The XRD pattern confirms the presence of rutile-type crystalline phase of SnO₂ nanoparticles formed along with the face-centered-cubic (fcc) metallic Au nanocrystals, after 24 h under solvothermal conditions. The TEM image clearly shows the formation of Au/SnO₂ nanocomposites. The gold nanoparticles formed are spherical in shape with the particle size of around 20 nm. The HRTEM image confirms the crystal structure of gold and SnO₂. The as-formed Au/SnO₂ nanocomposites are more stable and have potential applications as previously proposed.

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