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Sodium dodecyl sulfate assistant solvothermal Synthesis and Characterization of $Ag_2S/PbSO_4$ Nanocomposites

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ABSTRACT: Novel $Ag_2S/PbSO_4$ nanocomposites have been successfully synthesized by solvothermal process which propylene glycol used as a solvent. In this demonstration, sodium dodecyl sulfate (SDS) performs as a sulfur source to produce Ag2S/PbSO4, and also act as a surfactant and the samples were further studied by X-ray powder diffraction (XRD), surface morphological studies from scanning electron microscopy (SEM) and transmission electron microscopy (TEM), elemental estimation from Energy dispersive X-ray Spectroscopy (EDAX), also studied photoluminescence (PL). From photoluminescence studies, the absorption of nanocomposite with a band gap of 3.1 eV which depends on quantum confinement behavior. Besides, SEM investigation displayed the morphology and particle size of the final product, which were affected by the reaction time and surfactant.

Keywords: *$Ag_2S/PbSO_4$; Nanocomposites; Solvothermal;*

1. INTRODUCTION

The current interest of material scientists and engineers about the nanocomposites is due to their unique properties as minute nano size, larger surface area to volume ratio and study of crystlline nature. These prospects of nanocomposites lead to drastic improvement in their physical and chemical properties. Different nanocomposites, including hollow spheres Fe_2TiO_5/α-Fe_2O_3nanocomposite [1], carbon hollow spheres encapsulating silver nanoparticles [2], hollow silica/magnetic composite spheres [3], hollow zeolite spheres [4], Polyaniline (PANI)/Fe_3O_4 composite hollow spheres [5] and micro spheres $Mg_5(OH)_2(CO_3)_4{\cdot}4H_2O$ [6], CdS [6], Fe_3O_4 [8], $PbSO_4$ precipitated in a solution of polyethylenimine [9], rod-shaped $PbSO_4$ nanocrystals were prepared under the presence of poly sodium-p-styrene sulfonate (PSS) [10], lamellar mesostructures of nanocrystalline $PbSO_4$ prepared by hydrothermal treatment and lead sulfate films with two layer structure [11]. In present work the production of $Ag_2S/PbSO_4$ nanocomposites. Present method is done using two sulfide sources in presence of solvothermal route and absence of surfactants or additives. The morphological structure and particle size with change in the temperature and reaction time were also analyzed.

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Competing interests
The authors have declared that no competing interests exist.

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2. EXPERIMENTAL

2.1 Synthesis of Ag2S/PbSO4 nanocomposites

By the chemical approach, $Ag_2S/PbSO4$ nanocomposites were prepared in lab using the mixture of chemicals $AgNO_3$ (2mM), $Pb(NO_3)$ (1mM), and sodium dodecyl sulfate as sulfice source (2mM) with the molar ratio of Ag, Pb and S as 2:1:2 in 30ml propylene glycol and stirred for 2 hours and then was transferred to a Teflon autoclave of 100 ml capacity and the reaction was kept at 200°C for 4 hrs in an electric oven and the synthesis was done at various conditions , as shown in Table 1.

Table 1: $Ag_2S/PbSO_4$ nanocomposites reaction conditions.

Sample No	Sulfide Source	Temperature (°C)	Time (Min)
A1	SDS	200	4
A2	SDS	200	6
A3	SDS	200	10
A4	SDS	200	15

After the completion of reaction, the autoclave was cooled naturally to room temperature. The obtained yellow colour precipitate was processed for centrifugation and

washing with alcohol and distilled water and dried under vacuum at 75°C for 4 hrs. The characterization of the purity and morphology of the prepared $Ag_2S/PbSO_4$ nanocomposites was studied.

2.2 Characterization

The characterization of X-Ray diffractometer was done with Bruker D8 advance Cu Kα radiation (λ=1..54 Å). The morphology of the nanocomposite was studied by scanning electron microscope (SEM) (LEO 1455VP) and transmission electron microscope (TEM) with an accelerating voltage of 100kV (EM208 Philips). The Energy dispersive spectrum was done with XL30 Philips and the room temperature photoluminescence (PL) spectroscopy was observed with fluorescence spectrophotometer (Perkin-Elmer LS55).

3. RESULTS AND DISCUSSION

The obtained XRD spectra patterns (figure 1) were specified as $Ag_2S/PbSO_4$ nanocomposites, as it has the diffraction peaks of both Ag_2S and orthorhombic $PbSO_4$, as described by space group pbnm, JCPDS No.05-0577. In the same XRD graph a few diffraction peaks marked as "o" are of cubic Ag_2S as explained in JCPDS No.14-0072. The difference in mophology of $Ag_2S/PbSO_4$ nanocomposite with change in the reaction time and surfactant used for solvothermal method has been analyzed. $Ag_2S/PbSO_4$ nanocomposites synthesized from sodium dodecyl sulfate (SDS) and analyzed at different conditions.

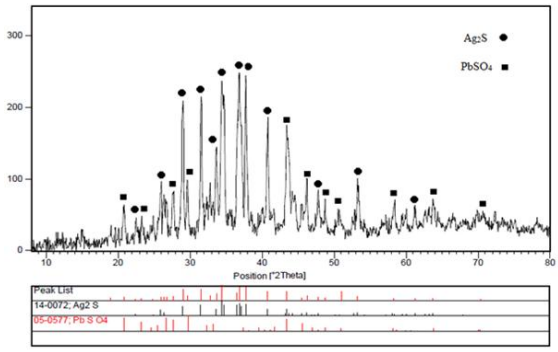

Fig. 1: XRD patterns of $Ag_2S/PbSO_4$ Nanocomposites.

The characteristics of synthesized $Ag_2S/PbSO_4$ nanocomposite as the size and morphology are determined by scanning electron microscopy (SEM), as shown in Figure. 2(a). The obtained nanocomposite have an average size of about 15-20 nm and only a very few negligible amount of larger grains coexist. To examine the effect of reaction time on the size of synthesized $Ag_2S/PbSO_4$ nanocomposites, Multiple synthesis were carried out on the basis of varying time, and the nanocomposites obtained from different time variables were characterized using SEM.

Figure 2b shows the SEM images of $Ag_2S/PbSO_4$ nanocomposite prepared with time of 6 h (sample A2), the product was mainly composed of more number of particles with average size of about 35 nm, and these $Ag_2S/PbSO_4$ nanocomposite with regular shapes were easily aggregated together. With the reaction progressing to 10 h, $Ag_2S/PbSO_4$ particle in the form of micro sphere, as shown in Figure 2c. If experiment involves, increased solvothermal treatment to 15 h while the temperature was still maintained at 200 °C, $Ag_2S/PbSO_4$nanocomposite with mean size of 10-15 nm were produced with the irregular shapes figure 2d..

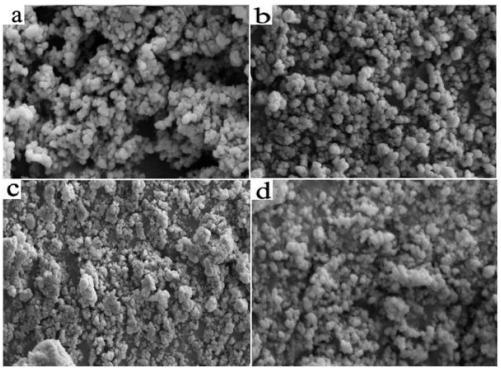

Fig. 2: SEM images of $Ag_2S/PbSO_4$ nanocomposites (a) A1 (b) A2 (c) A3 (d) A4

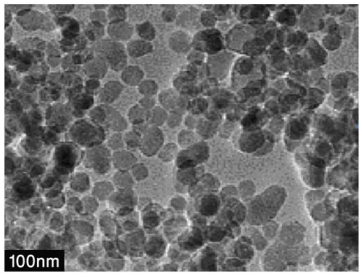

Fig .3: TEM image of$Ag_2S/PbSO_4$ nanocomposites (A2).

The morphological size and nanostructre of the $Ag_2S/PbSO_4$ nanocomposites were further examined using transmission electron microscope (TEM). TEM images of sample A2 is shown in figure 3. The $Ag_2S/PbSO_4$ nanocomposite consists of separated particles and the size is about 35 nm, almost consistent with observed SEM image sample A2,

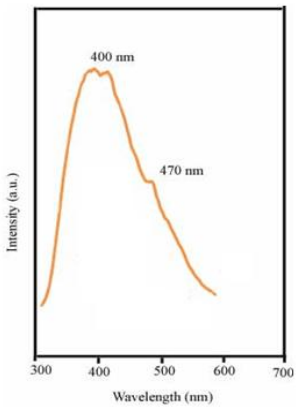

Fig. 4: Room temperature PL spectra of $Ag_2S/PbSO_4$nanocomposites sample no.A2

Photoluminescence (PL) measurement of $Ag_2S/PbSO_4$ nanocompositewas carried out at room temperature that laid out in figure 4. Photoluminescence spectrum of$Ag_2S/PbSO_4$ nanocomposite consists of two emission peaks at 400 and 470 nm, which are red-shift relative to

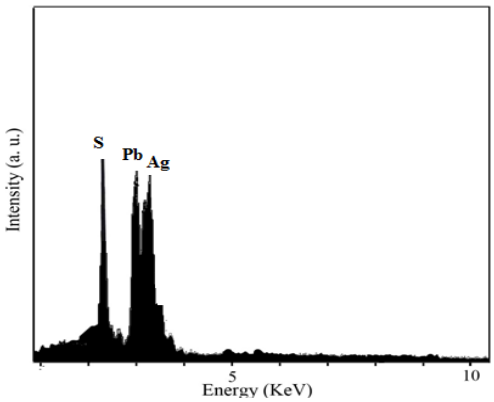

Fig. 5: EDS pattern of $Ag_2S/PbSO_4$ nanocomposites (sample no.A2).

bulk $PbSO_4$. These facts were caused by defects in the nanocomposites. To synthesize $Ag_2S/PbSO_4$ nanocomposites by the solvothermal reaction of $AgNO_3Pb(NO_3)_2$ and sodium dodecyl sulfate in propylene glycol at 200 °C for 4 h.The chemical composition and purity of synthesized nanocomposites were examined by EDS measurement (figure 5) which reveals the presence of Ag, Pb and S containing elements.

4. CONCLUSION

In the present experimental synthesis, sodium dodecyl sulfate (SDS) acted as surfactant and as well as sulfur source to prepare $Ag_2S/PbSO_4$ nanocomposites by solvothermal approach for reactions done at temperature 200°C for 4 hrs of time. The $Ag_2S/PbSO_4$ nanocomposites Photoluminescence (PL) emission was at approximately 400 nm. Silver nitrate ($AgNO_3$), Lead nitrate ($Pb(NO_3)_2$) and sodium dodecyl sulfate (SDS) were used as starting precursors, and propylene glycol was used as solvent agent. The size of the crystallites of the nanocomposites were around 30 nm. In this research, we have analyzed the different morphological and particle sizes of $Ag_2S/PbSO_4$ nanocomposites based on different reaction time prepared using solvothermal method. Finally, the obtained material $Ag_2S/PbSO_4$ nanocomposites were characterized using XRD, EDX, SEM and crystallite diameter (D_c) (calculated to be about 30 nm using the Scherer equation.)

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