



RESEARCH ARTICLE

SYNTHESIS AND CHARACTERIZATION OF PBS/PVA NANOCOMPOSITE FILM

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ABSTRACT

The nanocomposite film of lead sulphide nanoparticles and poly vinyl alcohol has been prepared by chemical synthesis method. Lead nitrate and sodium sulphide were used as lead (Pb^{2+}) and sulphur (S^{2-}) ion sources respectively. X-ray diffraction pattern revealed the cubic structure of PbS. Formation of the nanocomposite film is confirmed using the FTIR. Photoluminescence spectrum exhibited the two emission peaks corresponding to the blue-green and green emission of PbS. Electrical properties of PbS film were measured using impedance analyzer in the frequency range of 1 kHz to 1 MHz and in the temperature range of 303-348 K.

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INTRODUCTION

In recent years, hybrid nanocomposite materials based on organic polymers and inorganic semiconductor nanoparticles have attracted much more attention because of the good processability and solubility of organic polymers and the excellent optical, electronic and magnetic properties of inorganic semiconductor nanoparticles (Pomogailo *et al.*, 2000). Poly vinyl alcohol (PVA) is the most interesting organic polymer because of its variety of applications. It is a potential material having high dielectric strength, good charge storage capacity and dopant-dependent electrical and optical properties. PVA has carbon chain back bone with hydroxyl groups attached to methane carbons/these -OH groups can be a source of hydrogen bonding and hence assist the formation of polymer complexes. At room temperature conductivity of pure PVA is very low, but complexing it with dopants enhances its conductivity. PVA plays a vital role to enhance the good mechanical strength, long term temperature stability, optical, electrical, excellent film forming properties and surfactant materials of nanoparticles (Prajapati *et al.*, 2011). Lead sulphide (PbS) is an important IV-VI group of

semiconductor having cubic crystal structure and a narrow direct energy band gap of 0.41 eV at 300 K. PbS has a large exciton Bohr radius of 18 nm and it shows strong quantum confinement effect for relatively large sizes as compared to other semiconductors (Zhao *et al.*, 2005). Thus the band gap can be blue shifted from near infrared (IR) to the visible region by forming nanoparticles. It can be exhibited excellent optical, electrical properties and applications in nonlinear optical devices, electroluminescent devices and optical devices such as IR detectors, Pb^{2+} ion-selective sensors, solar cell, light emitting diodes and optical switches due to their exceptional third-order nonlinear optical properties. During the past years, various properties of PbS nanoparticles have been studied by various researchers for its applications point of view (Zhao *et al.*, 2005). However, a complete study of electrical parameters in a wide frequency and temperature range is still lacking. In this paper, we have completely studied the electrical properties of PbS nanoparticles in PVA polymer matrix. To best of our knowledge, this is the first time investigation on the electrical properties of PbS nanoparticles in the polymer composite. The structure, photoluminescence and impedance properties of nanocomposite film have been studied in detail.

Experimental section

In a typical experiment of synthesis PbS nanoparticles, lead nitrate (Solution A, in the 10 ml of distilled water) and sodium

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sulphide (Solution B, in the 10 ml of distilled water) were taken in the molar ratio of 1:1. Solution A was stirred vigorously by using magnetic stirrer. Then continuing same stirrer condition, solution B was added drop by drop to the above solution. After 2 hours, colour of the whole solution turned into dark brown. This indicates the formation of PbS. This reaction was continued upto 8 hours to form homogeneous solution. The resultant solution was washed several times with distilled water to remove unwanted impurities and dried in hot air oven at 80°C for 1 h. In order to form nanocomposite film, an appropriate quantities of PVA was dissolved in distilled water with stirring the solution at 70°C to complete dissolution, and then required quantity of PbS nanoparticles was added to the polymeric solution with continuous stirring and dispersed by ultrasonic agitation to ensure a homogeneous distribution of PbS nanoparticles. The solution was poured onto cleaned petri dish and allowed to evaporate the solvent at room temperature. After drying, the film was peeled from petri dish and kept in vacuum desiccator until use. The structural property of the nanocomposite film was studied with X-ray diffraction (XRD) using Rigaku Miniflex-II desktop x-ray diffractometer in the range of 10-60° using CuK α radiation ($\lambda=1.5406 \text{ \AA}$). The Fourier transform infrared (FTIR) spectrum was recorded for a nanocomposite film in the wave number range of 400-4000 cm^{-1} at room temperature using a Bruker Tensor 27 FTIR spectrophotometer with a resolution of 2 cm^{-1} . PL study has been carried out using the Perkin Elmer LS55 spectrometer in region of 400-700 nm at room temperature. The impedance properties were analyzed by N4L Phase Sensitive Millimeter interfaced with impedance analyzer in the frequency range of 1 kHz to 1 MHz over the temperature ranging from 303 to 348 K.

RESULTS AND DISCUSSION

XRD study

Fig. 1 shows the XRD pattern of PbS nanocomposite film. The diffraction peak at 34° is due to semicrystalline nature of PVA (Prajapati *et al.*, 2011). The peaks in 2 θ values equal to 26°, 30.2°, 43.2°, 51.1° and 53.5° which are assigned to the diffraction lines produced by (111), (200), (220), (311) and (222) crystalline planes of the fcc cubic phase rock salt structure of PbS. The lattice constant has been calculated to be about $a = 5.929 \text{ \AA}$, which is almost in agreement with the standard data (JCPDS Card File No. 5-592). The average crystallite size of PbS in composite film was calculated using the Debye-Scherrer equation and is found to be 13.2 nm.

FTIR study

FTIR spectra of pure PVA and PVA/PbS films are shown in Fig. 2. The spectrum of pure PVA film exhibits peaks at 3315, 2949, 1712, 1426, 1142, 918, 854 and 665 cm^{-1} which are assigned to O-H stretching, C-H stretching, C=O stretching, bending of CH₂, C-O stretching, CH₂ rocking, CH₂ stretching and O-H wagging respectively (Abdelaziz *et al.*, 2011). FTIR spectra of PbS nanocomposite film is found to be identical with that of the polymer composite, indicating the weak interaction between nanoparticles and polymeric matrix. This kind of interaction was observed earlier in the different polymer matrix with PbS nanoparticles (Kuljanin *et al.*, 2006).

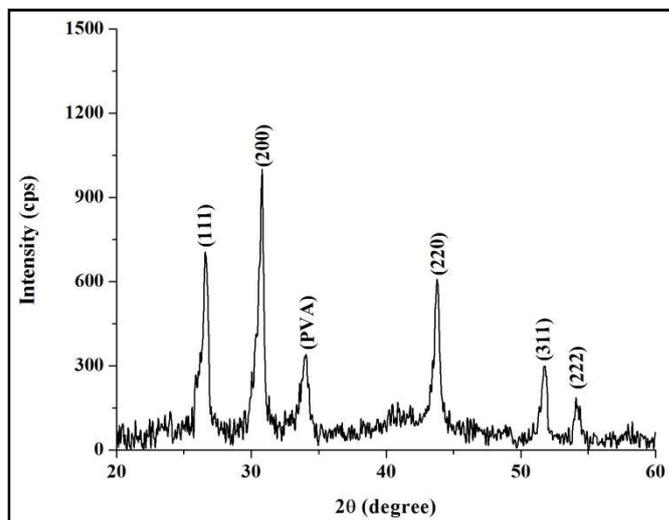


Fig. 1. XRD pattern of PVA/PbS nanocomposite film

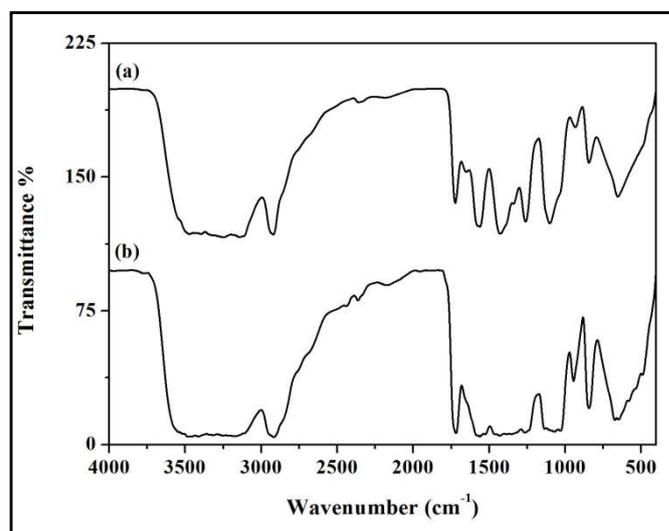


Fig. 2. FTIR spectra of (a) PVA film and (b) PVA/PbS film

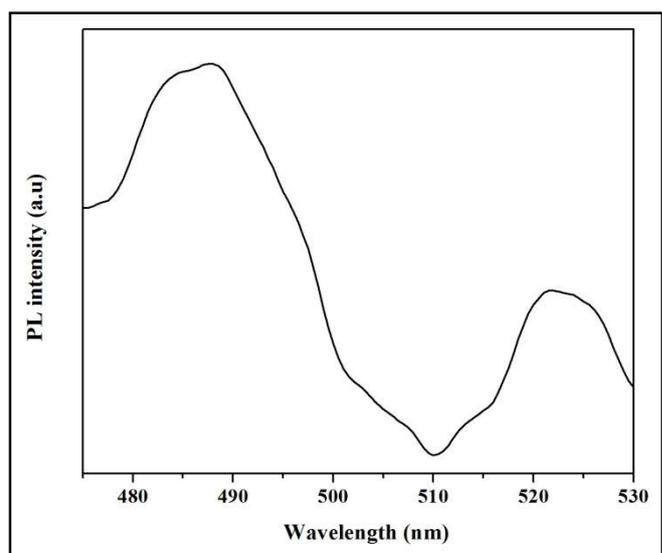


Fig. 3. PL spectrum of PVA/PbS nanocomposite film

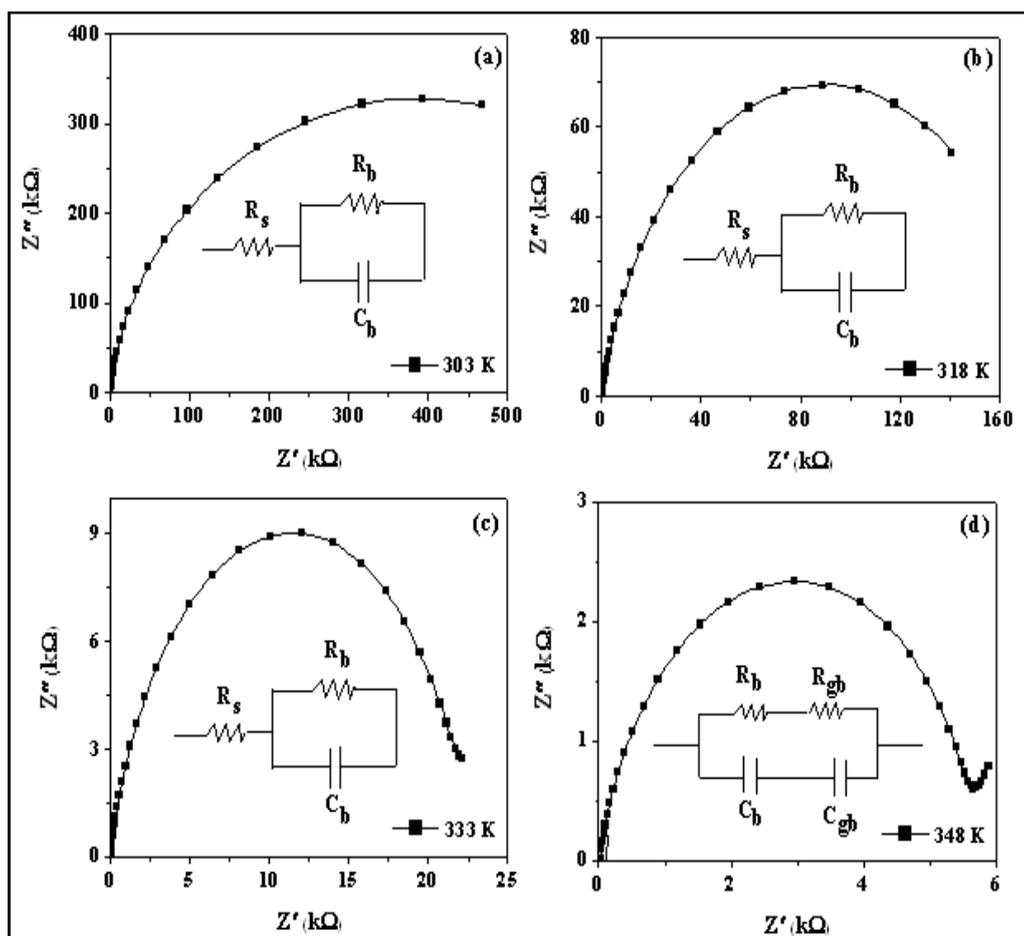


Fig. 4. Frequency dependence of Complex impedance plot of Cole-Cole plots of PVA/PbS nanocomposite film at different temperatures. Inset figure shows the fitted Cole-Cole plot and the equivalent circuit diagram of the same curve

Photoluminescence studies

PL study provides information of different energy states available between valence and conduction bands associated with band edge luminescence and recombination at defects. The PL spectrum of the as prepared- PbS film with an excitation wavelength 305 nm is shown in Fig. 3. It shows a broadened peak due to the small dimension of the nanoparticles in their wall, exhibiting a blue shift. It can be observed that the two emission peaks centered on 487 nm and 521 nm. The band present at 487 nm is known as blue-green emission peak and is ascribed to removal of sulfur anion vacancies. The second emission of PbS at about 521 nm is known as green band, which is observed from the recombination of electrons in singly occupied oxygen vacancies with photoexcited holes. Similar emission peak has been reported by earlier workers (Yongbin Zhao *et al.*, 2005; Zhao *et al.*, 2005).

Impedance analysis

Fig. 4 shows the frequency dependence of the complex impedance (Z) plot of PVA/PbS nanocomposite film at different temperatures. As the frequency increases the real part of impedance (Z') decreases where as imaginary part of impedance (Z'') decreases. This behavior continues up to a limited frequency for which Z'' attains a maximum value and

Z' intersects. For further increase in frequency both Z' and Z'' decrease which indicates existence of a relaxation phenomenon. All the semicircles exhibited some depressed arcs instead of semicircle centered on the real axis. This can be referred to as the non-Debye nature of relaxation. Moreover, the depression of the semicircle is considered as further evidence of polarization phenomena with a distribution of relaxation times. The Cole-Cole plot of the sample shows one impedance semicircular arc which represents the bulk properties of the grains. The equivalent circuit of PVA/PbS nanocomposite device consists of a parallel $R_b C_b$ (where R_b and C_b are bulk resistance and capacitance of the material) element with a series resistance R_s . It is also observed that the intersection of the semicircular arcs with real (Z') axis gives the bulk resistance (R_b) of the sample and is found to be decrease, indicated by a shift towards the origin with an increase in temperature. Furthermore, the plots at various temperatures demonstrate the pronounced temperature dependence of the device impedance in the low frequency range. Similar results have been reported for different nanocomposites by different authors (Mondal *et al.*, 2009; Mondal *et al.*, 2007).

Conclusion

In this work, we have successfully prepared PbS nanoparticles dispersed PVA polymer matrix using simple chemical method.

The crystal structure and particle size are determined by XRD. FTIR spectra confirmed the formations of PbS/PVA nanocomposite film. Photoluminescence spectrum showed the two emission peaks which are assigned to the blue-green and green emission of PbS in PVA film. The complex impedance plots shows non-Debye nature and distribution of relaxation times were confirmed by electric modulus analysis.

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