



Estimation of structural and mechanical properties of Cadmium Sulfide/PVA nanocomposite films



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ABSTRACT

Cadmium Sulfide (CdS) nanoparticles have been synthesized by hydrothermal method, and dispersed in varying amounts by weight, in Poly Vinyl Alcohol (PVA) matrix. Subsequently, PVA/CdS nanocomposite films have been characterized and analyzed to estimate CdS nanoparticles effect on the properties of PVA films. Both mechanical and structural properties have been considered for the analysis. From the FTIR (Fourier Transform Infrared Spectroscopy) analysis it's found that stretching of C-H, CO, COC and COS were observed at different peaks that may be due to different functional groups. X-ray diffraction (XRD) results showed that the matrix contain pure nano CdS particles. The images obtained by Scanning Electron Microscopy (SEM) showed the presence of varying degree of agglomerated CdS nanoparticles distributed homogeneously in the PVA/CdS nanocomposite. Thermal properties are found using DSC (Differential Scanning Calorimetry) was used to investigate. PVA/CdS samples were tested by Universal Testing Machine (UTM) and the results indicated improvement in mechanical properties when compared with neat PVA film, viz. Young's modulus (from 33.20 to 94.30 MPa), ultimate tensile stress (from 17.03 to 27.60 MPa), toughness (from 2.18 to 17.62 MPa) and flexural strength (from 4.43 to 7.66 Mpa) with increase in the CdS content in the nanocomposite film.

1. Introduction

Properties of nanomaterials differ substantially from other know materials based on two factors: increased relative surface area and Quantum effect. These factors can change and improve mechanical strength, reactivity, electrical, optical and magnetic characteristics of a material [1]. Cadmium sulfide is an important II–IV semiconducting material in view of its use as photo catalyst, non-linear optical material in solar cells, X-ray detectors and in display devices [2]. Nanoparticles have been synthesized by different methods: viz., solution combustion, hydrothermal, micro oven, sol-gel techniques, chemical precipitation and solve-thermal. Each method has its own merits and demerits such as requirement of high temperature, high energy input, special device or complexity of reactions and release of toxic reagents. In order to avoid complex reactions and toxic reagents, some researchers have been attempted to simplify the synthetic route for nanoparticles [3].

In recent years, research interest in the area of nanocomposites increased due to its uniqueness in properties. Polymers can be considered

as good materials for hosting metallic/semiconductor nanoparticles as the polymeric matrices have sufficient stiffness and strength along with other useful mechanical properties [4, 5]. The properties of both put together make their use as reinforcements for high structural strength applications is extremely attractive and great potential for advancement of material science technological research [6]. Hence nanocomposites have applications in diverse areas: like in catalysis, optics and electro-mechanical devices [7]. In general, reinforcing fillers found to alter the efficiency of polymer matrices. The efficiency enhances as the particle size of filler decreases. The increase in the ratio of surface area of dispersed nano-filler to the volume of polymer matrix increases the efficiency. This ratio also influences the strengthening performance of polymer composites. At higher values of dispersion ratio becomes more effective [8]. Hence, influence of dispersed nanoparticles on polymer matrix properties is investigated. PVA is easily soluble in water and has been chosen as a host for dispersing nano-fillers [9]. The studies on effect of various nano fillers embedded in a polymer matrix on mechanical, electrical and thermal properties are listed in Table 1.

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Table 1
Effect of nano fillers on the properties of polymer composite matrices.

Nano Filler	Matrix	Improved Property	Reference
Ag	PVA	Thermal Stability	[5]
Alumina	PVA	Dielectric Constant	[10]
CuO	PVA	AC Conductivity and Dielectric Loss	[11, 12]
		Tensile Strength, Flexural Strength, Toughness	
Al ₂ O ₃	Epoxy	Flexural Strength and Young's Modulus	[13]
Clay	HDPE	Dielectric Breakdown Strength	[14]
CdS	PVA	Dielectric Constant, AC Conductivity and Dielectric Loss	[15]
TiO ₂	PVA	Tensile strength and Young's modulus	[16]
Na ₂ ZrO ₃	PVA	AC Conductivity, Dielectric Constant and Dielectric Loss	[17]
ZnO	PVA	AC conductivity	[18]
Li ₂ ZrO ₃	PVA	Dielectric Constant and Dielectric Loss	[19]

This paper is aimed at synthesis of CdS, preparation of PVA/CdS composite, structural and mechanical properties estimation based on the dispersion of CdS nanoparticles in PVA polymer matrices. The influence of CdS nanoparticle addition on structural characteristics was analysed using Fourier infrared spectroscopy. The effect of CdS concentration on structural and morphological characteristics was analysed using X-Ray diffraction analysis (XRD) and Scanning Electron Microscope (SEM) and Differential Scanning Calorimetry (DSC). The mechanical properties of neat PVA films and PVA/CdS composites such as stress/strain relationships, toughness, flexural strength and other relevant properties were evaluated to analyse the effect of nanoparticle addition on the properties of PVA host.

2. Materials & methods

2.1. Reagents and materials

All reagents used without purification as they are analytical grade (AR). Cadmium nitrate, [Cd(NO₃)₂·4H₂O (≥99%)], Nice Chemicals Pvt. Ltd, Cochin, India, Thiourea [NH₂CSNH₂] (≥99%), L-valine [C₅H₁₁NO₂] BO grade and PVA [C₂H₄O]_n LR grade were procured from SD Fine-CHEM (SDFCL), Mumbai, India. The PVA was in powder form (molecular weight: 85000–124000 (86–89%). Purified water made from double distillation was used in the preparation of solutions, casting of films and other requirements.

2.2. Synthesis of CdS nanoparticles and characterization

CdS nanoparticles were prepared using hydrothermal method [15, 20, 21]. An aqueous solution was prepared by dissolving 3.08 g of Cadmium nitrate (Cd(NO₃)₂·4H₂O), 2.285 g of Thiourea (NH₂CSNH₂) and 0.3518 g of L-valine (C₅H₁₁NO₂) in 80 ml of double distilled water. Here, thiourea and L-valine act as fuel and stabilizer respectively. The fuel supplies required energy for the reaction. The solution is kept in Teflon lined stainless steel container shaken for 30 minutes and then placed in an autoclave at a temperature of 180–200 °C for 4 hours. Later it was cooled to ambient temperature. The product was washed thoroughly with water, ethanol and centrifuged. The residue was heated at 80 °C for an hour to produce nano powder of CdS. The overall chemical reaction can be represented as (reaction R1):



2.3. Casting of PVA/CdS nano polymer composite films

Solution intercalation film casting method is used for preparing the composites films consisting of polymer (PVA) and nano metal sulfide (CdS). Initially a solution is prepared by dissolving PVA powder in water by supplying heat and mixing for 4hrs at 80 °C (±3 °C). CdS powder is

added to this solution and the resultant suspension is made homogeneous (uniform distribution and minimal agglomeration of nanomaterial in solution) by ultra-sonication for about 45 minutes at 85 °C (±3 °C). The suspensions containing 7.5% PVA are used for all batches. The solution immediately transferred into an already cleaned and dried glass mold. The mold is dried in dust-free atmosphere at 30±3 °C for about 2 days. The dry films were found to have evenly dispersed CdS nanoparticles devoid of any air bubbles. Different films were casted using solutions containing varying amounts of CdS nanoparticles (between 0.5 to 2% CdS by weight). The thickness of casted films, measured by LCD digital vernier calipers, is in the range of 0.18–0.22 mm [15].

2.4. Measurements

The FTIR spectra (Spectral range: 4000–400 cm⁻¹) of all dried composite films were taken with a resolution of 4 cm⁻¹ using transmittance mode. Bruker's D-8 XRD was used for analysis (Source of X-ray: Ni-Cu-Kα radiation at 40 kV and 30 mA; Range: 0–80° at 8° min⁻¹ speed). Surface images were recorded using scanning electron microscopy (Model: JSM-5910-JEOL) operated at 5 kV. NETZSCH DSC 200F3 is used for thermal analysis, carried out in inert (N₂) atmosphere between 30 to 250 °C. Both heating and cooling rates were maintained at 10 °C min⁻¹ during measurement. Mechanical properties were estimated using Universal Testing Machine (KIPL-PC 2000; Error range: ±0.2 MPa; Test speed for gripping surface: 10 mm/min; Load cell: 0.2 kN).

3. Results and discussion

3.1. FTIR analysis

Possible interactions between PVA and CdS can be understood by FTIR analysis. The spectra (Fig. 1) show peaks roughly around at 3270, 2920, 1720, 1240 and 1085 cm⁻¹. A small peak at 835 and few peaks are also found between 400–515 cm⁻¹. The broad peak around 3270 cm⁻¹ indicates stretching of hydroxyl groups (O-H), and peaks at 2920 cm⁻¹ is due to C-H stretching vibration is asymmetric. The peaks observed at 1720 cm⁻¹ corresponding to C=O out-of-phase stretching (of vinyl acetate group of PVA) and CH₄ in-plane deformation respectively. Peak found at 1240 cm⁻¹ related to out-of-phase stretching of C-O-C. Peak at 1085 cm⁻¹ corresponds to C-O-S stretching and also indicates the C-O stretch of secondary alcoholic groups. Out-of-phase stretching observed at 835 cm⁻¹ was due to sulfate ions linked with water molecules by hydrogen bonding [22]. The strong absorption bands observed between 460–485 cm⁻¹ is due to the CdS stretch vibrations [23]. The observed broad peak at 3270 cm⁻¹ is purely due to the presence of moisture adsorbed. It is evident that alkyl groups are capped on CdS from the peak observed at 2920 cm⁻¹. No additional peaks are found in IR spectra but a small shift in position of peaks noticed (which is not considerable) indicating physical interaction among hydroxyl groups and nano CdS present in film. These hydroxyl groups take part in chelating reactions with nano CdS particles and form charge-transfer complexes.

3.2. X-ray diffraction analysis

The XRD Patterns of nano CdS is shown in Fig. 2. On comparison with standard patterns of JCPDS (Number: 41-4019) [20], it revealed that the synthesised CdS has hexagonal crystalline structure, as several sharp peaks are seen at 2θ values of 24.98, 26.5, 28.14, 31.8, 43.8, 48.2 and 51.9°. Variation in the solvent quantity during the synthesis had prominent effect on growth of crystals [24]. As the solvent quantity is not varied while preparing the samples in present study such preferential growth was not observed.

Patterns of XRD for pure PVA and PVA/CdS Composite films are shown in Fig. 3. Fig. 3(a) for pure PVA and Fig. 3 (b)–(e) for different CdS filler concentration between 0.5 and 2 wt%. For all compositions the peak was obtained at 2θ ~ 19.4° [25], which correspond to crystal plane

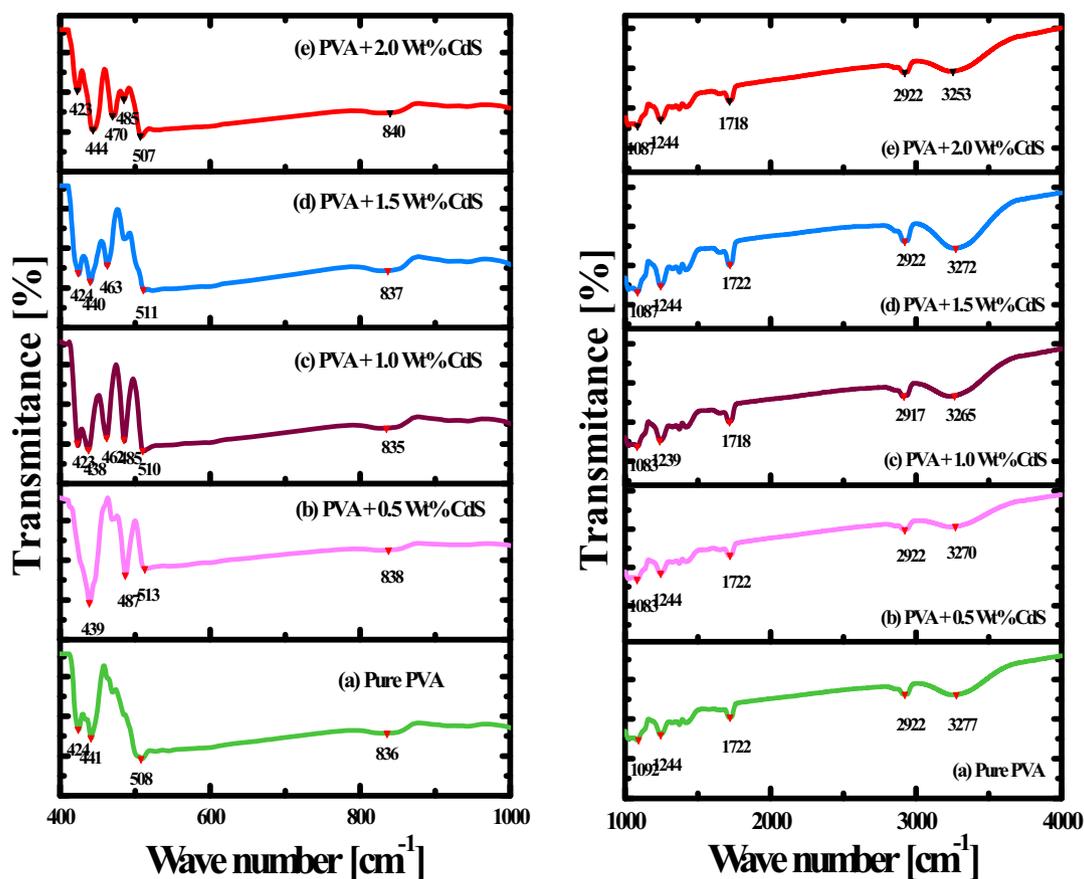


Fig. 1. FTIR of PVA films loaded with nano CdS Concentration of CdS in weight%: (a) 0.0 (b) 0.5 (c) 1.0 (d) 1.5 and (e) 2; Wave number in cm^{-1} : (i) 400–1000 (ii) 1000–4000.

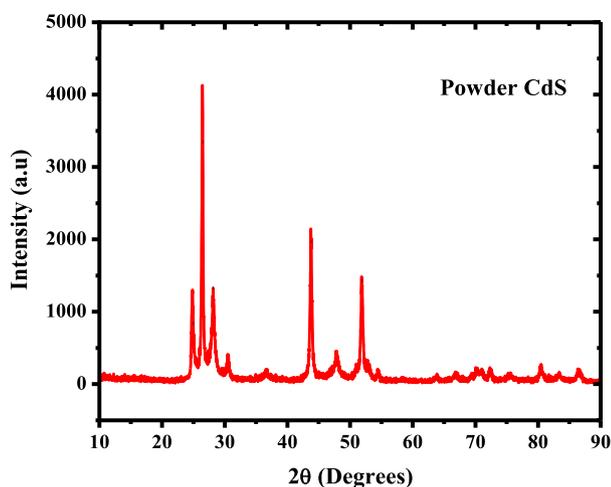


Fig. 2. XRD patterns of pure nano CdS.

(1 0 1) for PVA indicating its semi-crystalline nature. This is result of strong interaction between the molecules in the polymer network. The mean crystalline size of synthesized nano CdS was calculated using Debye-Scherrer's equation, was found to be 37.68 nm. Broadening or shifting of peak (at $2\theta \sim 19.4^\circ$) was present in the profiles of any film but small newer peaks at $24.98, 26.4, 28.16, 30.65, 44.33, 47.92, 51.81$ and 54.48° were appeared, indicating clearly the existence of nano CdS inside the polymer composite matrices.

3.3. Images from SEM analysis

The SEM images shown in Figs. 4 and 5 explain surface morphology of CdS nanomaterial and PVA/CdS nanocomposite films prepared at different concentration of CdS nano filler (0–2%) respectively. It can be observed from the images that the CdS nanoparticles have acicular (Needle like components), porous, agglomerated structure with a particle size below 80 nm and in good agreement with reported images elsewhere [1, 26]. The nonuniform size distribution may be partly due to agglomeration that takes place to minimise higher surface energy possessed by individual nano-sized particles.

The white spots in Fig. 5 represents the agglomeration of few CdS nanoparticles, found at all concentrations of CdS. The agglomerated CdS nanoparticles distributed homogeneously at lower concentrations. In Fig. 5d–e, the agglomeration leads to big masses of nanoparticles. Increase in agglomeration or decrease in dispersion was observed with increase in the filler concentration. As particle is in nano range Vander Waals force of attraction is more, resulting in agglomeration. Due to agglomeration increase in particle sizes were observed with increase in the concentration of filler. The increase in particle size may be attribute to decrease in interparticle distance as number of particles increase with increase in the nano CdS filler concentration.

3.4. Thermal characteristics

Thermal characteristics of casted films of PVA/CdS are reported in our earlier studies on electrical characteristics of PVA/CdS composite films analyzed by DSC [15, 27], in which the investigation of CdS filler effect on thermal properties was carried out. The results are summarized in Table 2.

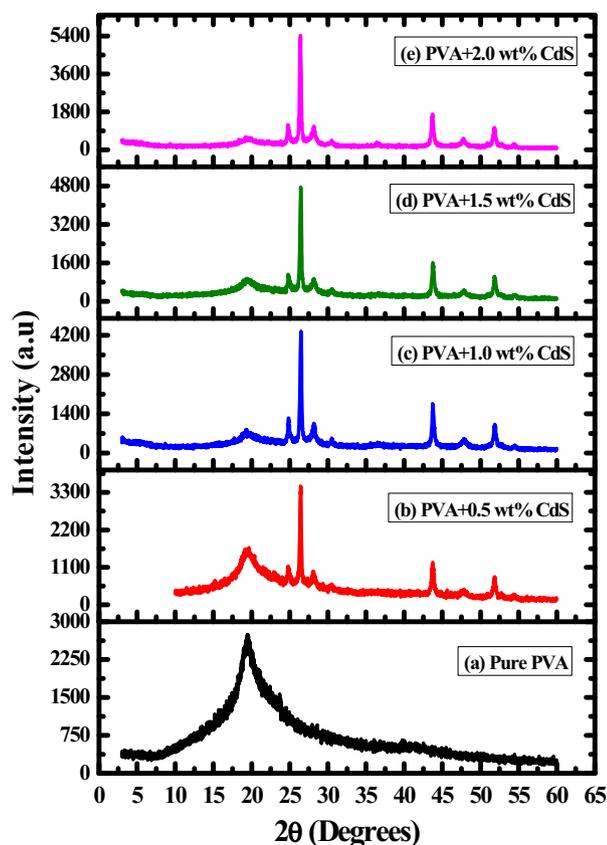


Fig. 3. XRD of nano CdS loaded PVA films Concentration of CdS in weight%: (a) 0.0 (b) 0.5 (c) 1.0 (d) 1.5 and (e) 2.

The study indicated that glass transition temperature (T_g), Melting temperature (T_m), Crystallization temperature decreased with increase in the CdS filler concentration. CdS loading has no major effect on crystallinity (melting enthalpies almost were unchanged), it can be concluded that nano CdS presence prevents crystal thickening.

3.5. Mechanical properties of nanocomposites

The mechanical properties of pure PVA and PVA/CdS composite films were estimated using UTM (KIPL-PC 2000; Error range: ± 0.2 MPa; Test speed for gripping surface: 10 mm/min; Load cell: 0.2 kN) as per ASTM D 638 (Standard Test method for Tensile properties of plastics). The thickness and width of films were in the range of 0.18–0.22 mm and 17–22 mm respectively. Mechanical properties of nano-composites have high reinforcement due to dispersion, larger surface area and intermolecular interactions of different functional groups present in the matrix of composites [28]. Various mechanical properties measured in this study to show effect of filler content in polymer matrix are explained below.

3.5.1. Stress–strain curves

Tensile tests were performed to break the samples of PVA and PVA/CdS nano composites. Fig. 6 shows in stress/strain curves of neat PVA and prepared nano-composite film. Specific tendency was seen for all films: the stress level increased by addition of CdS nanoparticles representing their role in reinforcement. The composites show signs of linear stress–strain character up to failure, plastic deformation and also confirm similar curve shape for plain and nano filler composites [29]. An improvement in tensile strength and modulus was observed for all samples.

3.5.2. Effect of filler on Young's modulus

Young's moduli (E) of PVA/CdS nano-composites are presented in

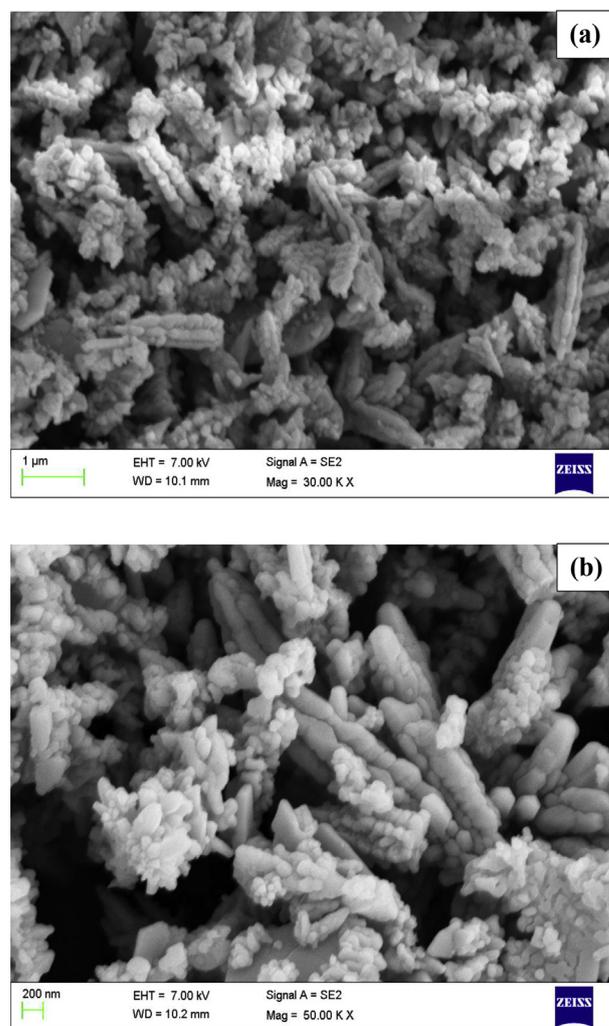


Fig. 4. SEM image CdS nanoparticles taken at micrometre (a) 1 μ m and (b) 200 nm scale.

Table 3 and Fig. 7. It is observed that Young's modulus increased with increase in the content of nano CdS but with a small deviation at higher loading of 2.0 wt%. The largest value of E (94.3MPa) was obtained for 1.5 wt% CdS. Hence it is suggested that nanoparticles can be incorporated into a PVA matrix to improve its stiffness causing a dilution effect, i.e. the volume fraction of PVA matrix decreased with increasing filler loading, which would lead to a decrease of cross-linking of composites. In general, a major improvement in mechanical properties is credited to the strong interaction between CdS nanoparticles and PVA matrix.

3.5.3. Ultimate tensile strength

Fig. 8 shows the change in ultimate tensile strength as function of CdS filler content. The ultimate tensile strength at break observed was varied with increasing in filler contents and the similar trend was reported elsewhere [30]. The observed values show an improvement in tensile strength of polymer nanocomposites compared to neat PVA for all concentrations of filler. Hence it can be said that the mechanical properties of composites get higher reinforcement due to dispersion of nano CdS particles in the matrix and it is attributed to greater surface area and numerous interactions involving different functional groups of matrix and filler in the composites [28].

Increasing CdS loading in the polymer composite, tensile strength first rose sharply and also increased at higher CdS loadings. The most important progress is obtained at a loading with 0.5, and 1.0 wt% of CdS films reaching 21.97 (29% increase) and 27 MPa (58.54% increase)

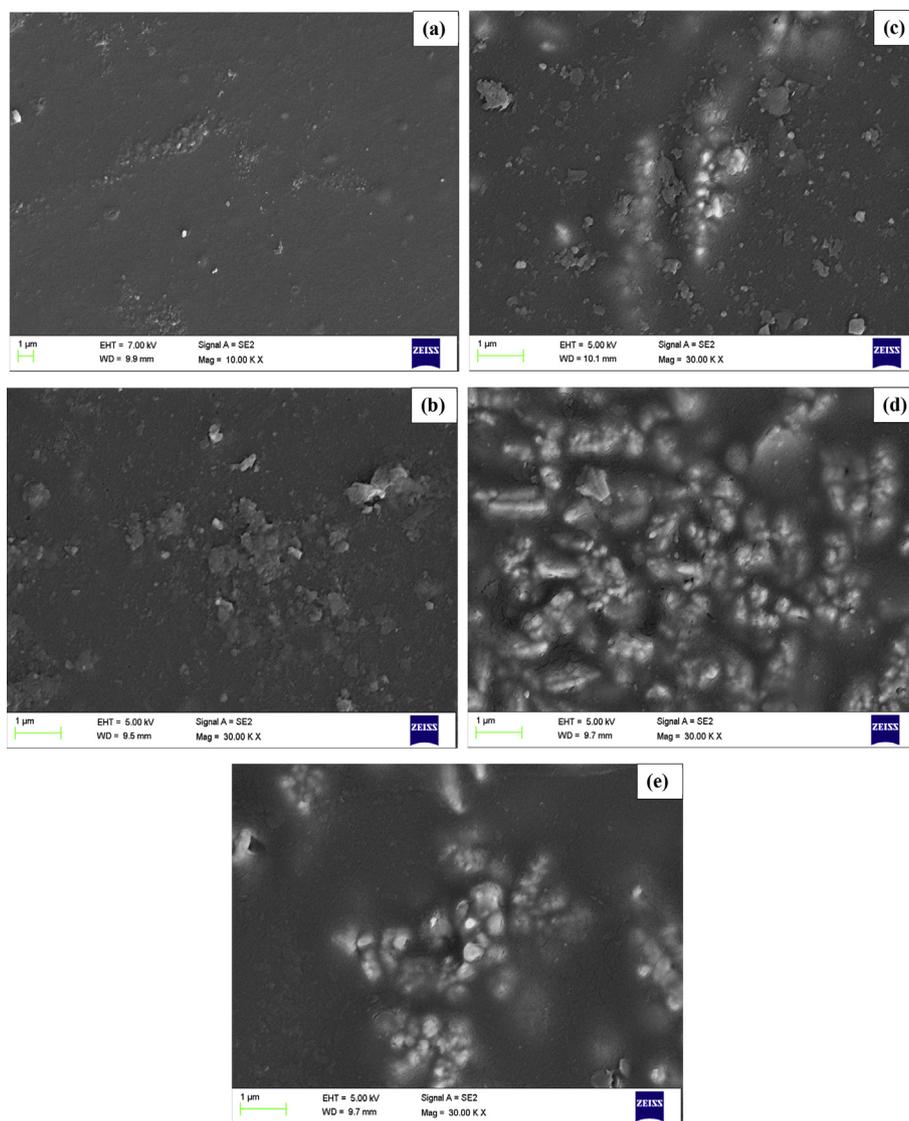


Fig. 5. SEM photomicrographs of nano CdS loaded PVA films Concentration of CdS in weight%: (a) 0.0 (b) 0.5 (c) 1.0 (d) 1.5 and (e) 2.

Table 2
Range of values for measured thermal properties of PVA/CdS nanocomposites [15].

Property	Units	CdS content (wt.%)					
		0	0.5	1.0	1.5	2.0	
CdS Content in PVA (wt.%)	-	0	0.5	1.0	1.5	2.0	
Glass transition Temperature, Melting Temperature,	T_g	°C	81.6	71.3	68.4	71.2	69.2
Heat of Fusion, Percentage Crystallinity, Crystallization Temperature,	T_m	°C	224.3	189.1	190.2	191.5	190.0
	ΔH_m	J/g	40.54	40.89	41.49	37.29	35.72
	$\%X_c$	%	4.39	5.768	4.572	4.408	4.171
	T_c	°C	189.8	95.2	118.5	109.9	118.6

tensile strength respectively when compared to neat PVA. Further increase in CdS loading has no effect in ultimate tensile strength.

3.5.4. Toughness

Toughness is an estimate of the amount of energy consumed before the material fractures or is measure of the amount of energy essential to fracture a sample. Energy consumed is work done, product of force and

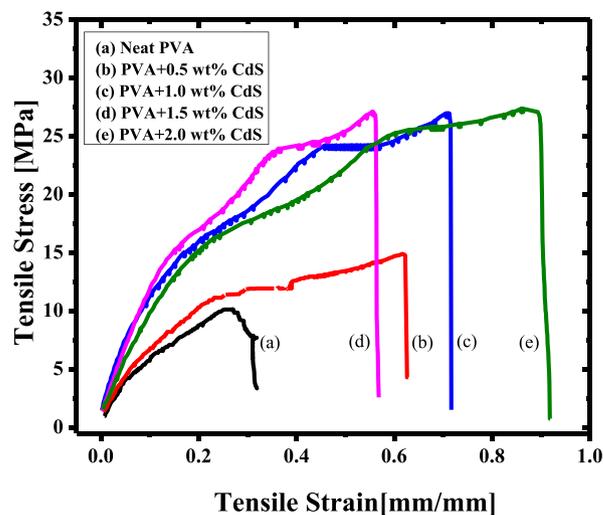


Fig. 6. Stress/strain variation as a function of nano CdS loaded PVA films Concentration of CdS in weight%: (a) 0.0 (b) 0.5 (c) 1.0 (d) 1.5 and (e) 2.

Table 3
Effect of nano CdS filler concentration on mechanical properties.

CdS content (wt %)	Young's Modulus (MPa)	% Synergy in Young's Modulus	Toughness (MPa)	% Synergy in toughness	Peak load at break point (N)	UTS (MPa)	Flexural Strength (MPa)
0.0*	33.20	-	2.18	-	17.7	17.03	4.43
0.5	43.60	31.32	6.67	205.96	21.8	21.97	5.45
1.0	75.50	127.40	13.64	525.69	29	27.00	6.59
1.5	94.30	184.04	10.54	383.49	21.6	27.10	6.75
2.0	91.00	174.10	17.62	708.26	24.5	27.60	7.66

*Corresponds to neat PVA.

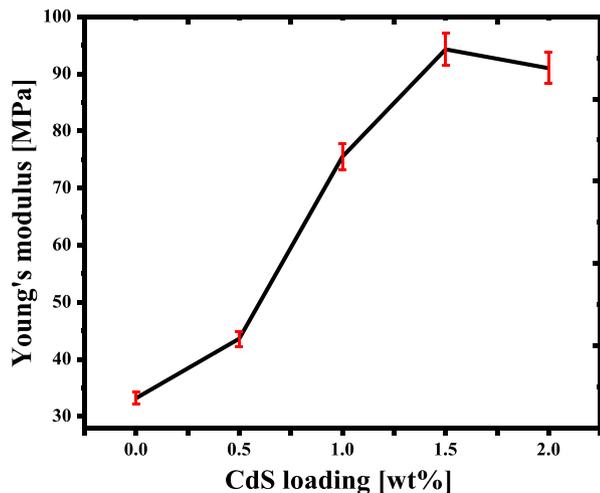


Fig. 7. Variation of Young's modulus versus nano CdS content in PVA (by wt%).

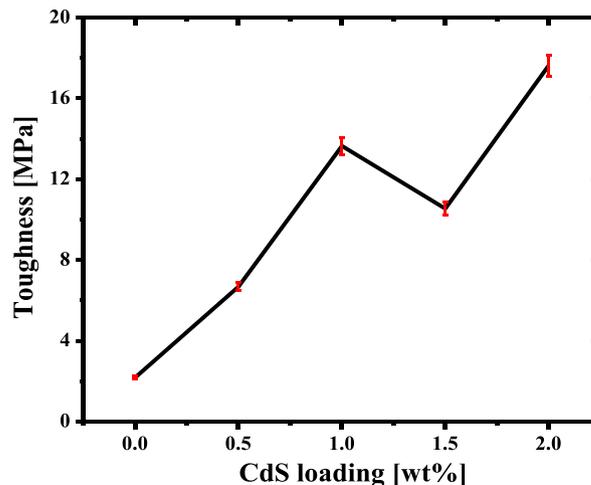


Fig. 9. Variation of toughness versus nano CdS content in PVA (by wt%).

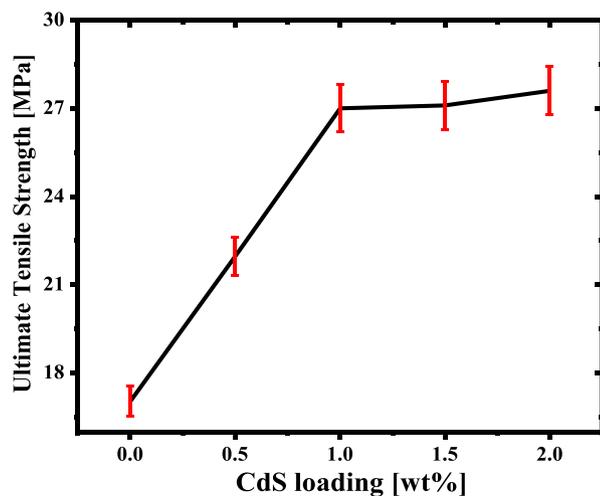


Fig. 8. Variation of ultimate tensile strength versus nano CdS content in PVA (by wt%).

distance. To compute toughness, true stress and true strain are used. Significant improvement in toughness nano-composite films of PVA/CdS was observed in present study is tabulated in Table 3 and represented by Fig. 9. The nano CdS/PVA film containing 2% filler showed largest improvement in toughness (by 710%) compared to that of pure PVA film. This improved toughness may be attributed to the morphological and structural changes stimulated as result of embedment of nanoparticles. This study indicated the maximum toughness of composite film containing 2% nano CdS.

3.5.5. Flexural strength measurement

Flexural strength (σ , transverse/modulus of rupture or bend strength), defined as stress at which yield in flexure test is possible in a material and calculated by the Eq. (1).

$$\sigma = \frac{FL}{bd^2} \tag{1}$$

Where: F is load at N (fracture point), L is length if support span, b is width and d is thickness.

Estimated Flexural properties from Eq. (1) of neat PVA and CdS containing PVA composites tabulated in Table 3 and are presented in Fig. 10. The results indicated an increase in flexural strength of polymer composites from 4.43 to 7.66 MPa with gradual increase in CdS content from 0 to 2%. The improvement in flexural strength may be due to higher specific surface of nanoparticles, dispersion and aspect ratio of filler and crystalline clusters that have certain stiffness. The mechanical deformation helps to prevent polymer matrix from fracturing due to the interaction at filler-matrix interface.

4. Conclusions

The addition of CdS in PVA host has strong influence on different properties of the nanocomposite. FTIR spectra shows strong interaction between hydroxyl groups and nano CdS. The crystallinity of CdS and PVA/CdS was confirmed by XRD. Uniform distribution of CdS in PVA matrix was observed till 1.0 wt.% loading, further increase in filler concentration resulted in agglomeration and the same was confirmed from the SEM images. The results of present work proved that the mechanical properties of PVA/CdS nanocomposites can be enhanced by incorporating CdS nanoparticles into the matrix. CdS loading of 2.0 wt.% has enhanced ultimate tensile strength (by 50%), flexural strength (by 53%) and toughness (by 173%) when compared to neat PVA. Present

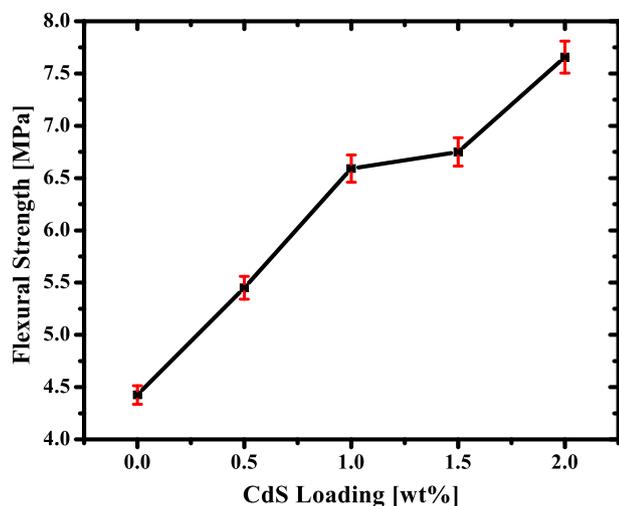


Fig. 10. Variation of flexural strength versus nano CdS content in PVA (in wt%).

results suggest that the CdS/PVA nano films may be potential candidates for nanotechnology in future where these properties predominantly influence the performance and can be further explored based on the need of application.

Declarations

Author contribution statement

G M Madhu: Conceived and designed the experiments; Analyzed and interpreted the data; Contributed reagents, materials, analysis tools or data; Wrote the paper.

Jammula Koteswararao: Conceived and designed the experiments; Performed the experiments; Contributed reagents, materials, analysis tools or data; Wrote the paper.

Vuppala Venkatesham: Performed the experiments; Wrote the paper.

Suggala Venkata Satyanarayana: Analyzed and interpreted the data.

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Competing interest statement

The authors declare no conflict of interest.

Additional information

No additional information is available for this paper.

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