

Effect of temperature on structural, morphological, and optical properties of Mn-doped ZnTe nanoparticles

KAPIL*, PUJARANI PARIDA*, JAYASHREE PATRA, VIRENDRA KUMAR VERMA*

Department of Physics, School of Advanced Sciences, VIT-AP University, Amaravati, Andhra Pradesh-522241, India

The Mn-doped ZnTe nanoparticles were synthesized by solid-state reaction, and the effects of Mn-doping on their physical properties were studied. Fourier-transform infrared spectroscopy spectra reveal a slight band shift from Zn-Te-Mn stretching. UV-Visible studies reveal that the band gap of ZnTe (2.11 eV) narrows to 2.06 eV with Mn doping, while higher calcination temperatures increase the band gap again. The band gap of ZnTe can be tuned through Mn doping and calcination temperature, enabling application-specific optimization. These results emphasize the role of Mn doping in tailoring the properties of ZnTe and enhancing its potential for visible-light photocatalysis and optoelectronic devices.

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1. Introduction

The semiconductor compound comprises elements from Group II (metals) and Group VI (chalcogens) of the periodic table. These materials have attracted significant attention in both fundamental research and practical applications. The optical and electrical characteristics of the II–VI compound semiconductors, as well as their large direct band gaps, are well-known [1]. Compounds such as ZnO, ZnS, ZnSe, ZnTe, CdO, CdS, CdSe, and CdTe, belonging to Groups IIB and VI, are widely utilized in solar cells, semiconductor lasers, light-emitting diodes, visible light photodetectors, nonlinear optical materials, magneto-optical devices, radiation detectors, and various optoelectronic applications [2–5]. These materials are crucial for photovoltaic conversion and optoelectronic applications, competing with silicon and other materials. Zinc telluride (ZnTe) is one such II–VI semiconductor that garnered considerable interest recently due to its potential applications in solar energy and optics. ZnTe has a direct bandgap of about 2.26 eV at room temperature and high absorption coefficients in the visible spectrum, making it ideal for use in light-emitting diodes, solar cells, photodetectors, and nonlinear optical devices [6]. Its compatibility with other II–VI and III–V compounds and its relatively high refractive index further improve its adaptability in device fabrication. Transition metals (Mn, Cr, Fe, Ni, Co, etc.) can be substituted into non-magnetic semiconductors (II–VI, II–V, III–V, and IV–VI) to create a new class of semiconducting materials called dilute magnetic semiconductors (DMS) [7–10], which are the most promising materials for use in spintronic and photocatalytic devices [11,12]. Magnetic ions of II–VI compound DMSs, including ZnTe, ZnSe, CdSe, and CdTe, are readily absorbed with +2 oxidation states. Mn-doped ZnTe has emerged as a promising ternary material for photovoltaic

applications. In recent years, conductive oxides have garnered significant attention due to their versatile applications in semiconductor devices, particularly in gas sensing [13,14]. Among the II–VI semiconductor family, Mn-doped ZnTe stands out as a highly attractive material owing to its exceptional electrical, optical, and piezoelectric properties [15–17]. Khan *et al.* [15,16] observed that Mn doping into ZnTe tailors the intra-band *d-d* transitions and defect-states, band gap, confirming it as a strong potential for optoelectronic applications. This compound exhibits a wide energy band gap, high excitonic binding energy, and significant photoconductivity, making it a compelling candidate for advanced technological applications. The properties and applications underscore the potential of Mn-doped ZnTe as a cost-effective and promising material for next-generation semiconductor technologies. Applying the appropriate doping elements in ZnTe is also crucial for customization of the intrinsic physical features, including luminescence and electrical characteristics. Particle size, crystallinity, and defect concentration all have a significant impact on the structural, optical, and electronic properties of ZnTe, which can be modified by the dopants.

Several methods have been used to prepare ZnTe-based DMS compounds, such as the chemical vapour deposition [18], hydrothermal method [19], sol-gel method [20], and spray pyrolysis [21] using different precursors. This study employed a freestanding solid-state reaction (SSR) method to synthesize Mn-doped ZnTe nanoparticles (NPs), offering a distinct advantage over conventional chemical preparation techniques. This approach eliminates potential contamination sources, thereby minimizing the influence of magnetic impurities. The resulting NPs underwent comprehensive characterization to assess their structural, morphological, optical, and functional properties, including the presence of vibrational groups, using powder X-ray diffraction (XRD), scanning electron microscopy (SEM),

*Equal contribution

UV-Visible (UV-Vis) spectroscopy, and Fourier-transform infrared spectroscopy (FTIR) techniques.

2. Experimental details

2.1. Experimental procedure

High-purity chemical reagents, manganese (II) oxide (MnO, 99%), and zinc telluride (ZnTe, 99.9%), were sourced from Nanoshell to synthesize pure and doped ZnTe. Using the SSR method, the following NPs were prepared: pure ZnTe (labeled S1), Zn_{0.95}Mn_{0.05}Te (labeled S2), Zn_{0.95}Mn_{0.05}Te calcinated at 100 °C (labeled S3), and Zn_{0.95}Mn_{0.05}Te calcinated at 250 °C (labeled S4). The precursor materials were ground for 1-2 hours to achieve a homogeneous mixture. Then the Zn_{0.95}Mn_{0.05}Te sample was calcinated at 100 °C and 250 °C for 2 hours in a furnace. After cooling to room temperature, the samples were thoroughly crushed and prepared for characterization.

2.2. Characterization

The structural characteristics of pure and doped ZnTe NPs were investigated using XRD analysis, conducted on a Rigaku Miniflex 600G diffractometer equipped with a point detector and utilizing Cu K α radiation ($\lambda = 1.5406$ Å). Data were collected over a 2θ range of 10° to 90°. The surface morphology of the NPs was studied using SEM on a ZEISS EVO 15 microscope. The FTIR spectra were obtained using a Cary 630 FTIR spectrometer in the 400-4000 cm⁻¹ wavenumber range. The optical properties were studied using UV-Vis DRS on a PerkinElmer Lambda 365 spectrophotometer.

3. Results and discussion

3.1. X-ray diffraction analysis

The XRD patterns of Mn-doped ZnTe NPs were obtained by adding 5% Mn dopant into pure ZnTe, and 5% Mn-doped ZnTe was calcined at different temperatures, specifically 100 °C and 250 °C. The XRD spectra of the pure ZnTe and doped ZnTe powder samples are displayed in Fig. 1(c). The XRD patterns of both undoped and doped powders indicated that the samples exhibited a cubic crystal structure of space group F-43m, as shown in Fig. 1(a). The main diffraction peaks of this phase are found at 25.23°, 41.87°, and 49.54°, which correspond to (111), (220), and (311) reflection planes for S1 samples, respectively. The 2θ degrees of the ZnTe (220) diffraction peak are 41.87°, 41.85°, 41.89°, and 41.90° for the S1, S2, S3, and S4 samples, respectively. When Mn is doped into ZnTe, the XRD peak slightly shifts towards a lower angle, but by heating the sample at different temperatures, the peak shifts towards a higher angle, which confirms that Mn is incorporated into ZnTe. The S3 and S4 samples, calcinated at 100 °C and 250 °C, exhibited the same peaks without any secondary phases in Fig. 1(b). XRD analysis revealed no

secondary Mn peaks, with all diffraction peaks of both pure and Mn-doped ZnTe powders aligning with the ZnTe JCPDS standard pattern. This confirms that Mn ions substitute into ZnTe lattice sites without introducing interstitial defects. The tetrahedral coordination and larger effective ionic radii of Mn²⁺ (0.66 Å) and Zn²⁺ (0.60 Å) indicate that Mn²⁺ substitutes for Zn²⁺ in the lattice. The peak sharpness shows a slight increase from S1 to S2, suggesting that Mn doping may initially promote crystal growth or reduce lattice strain, leading to a modest increase in crystallite size. Further sharpening is observed from S2 to S3 and S4, driven by atomic rearrangement and grain growth facilitated by higher calcination temperatures. S4, with the highest calcination temperature of 250 °C, displays the most significant sharpness, indicating the largest crystallite size among the samples, suggesting changes in grain size, potentially encompassing multiple crystallites. This behaviour is attributed to a refinement in crystallite size with increasing Mn content and temperature. A similar type of observation was also reported in Tang *et al.* [19], which confirms the single zinc blende structure in the Mn-doped ZnTe sample, with no secondary phase, and found that crystallinity increases with temperature. The diffraction patterns also indicate that the films are polycrystalline, with the (111) and (220) planes exhibiting the highest intensity. These findings are consistent with prior studies, showing a strong correlation with previously reported diffraction patterns [22]. The crystalline sizes for all samples are calculated and shown in Table 2 using the intense (220) peak. The sharp and narrow peak intensity of the XRD patterns indicated that the prepared sample has a high quantity and good crystallinity. After doping, there is no secondary peak, indicating that Mn is substitutionally incorporated into the Zn site within the ZnTe lattice, or perhaps the lattice accommodates the substitution without forming a new phase. Table 1 presents the calculated lattice parameters of Mn-doped ZnTe samples. By applying Bragg's law to XRD data, one can determine d-spacing and lattice parameters. The lattice parameters of the Mn-doped ZnTe sample align precisely with those reported in the JCPDS File. For the S4 sample, the intensity slightly decreased compared to undoped ZnTe. This may be due to the temperature effect, as the sample was calcinated at 250 °C. The lattice parameter decreases from sample S1 to sample S4, which may indicate an increase in the chemical pressure within the structure [23]. The XRD analysis is a simple and powerful method to calculate the crystalline size and lattice strain. The crystalline size can be calculated from the XRD peak of (220) by using the Debye-Scherrer equation (1).

$$D = \frac{k\lambda}{\beta_{hkl} \cos\theta} \quad (1)$$

where k is a Shape factor (~ 0.9), D is the Crystalline size, λ is the Wavelength of the X-ray used ($\lambda = 0.1540$ nm), β is the full width at half maximum (FWHM) (in radians), and θ is the Bragg angle. The value of crystalline size and strain increases by adding the dopant and by increasing the temperature in the pure ZnTe sample, forming better

crystallinity in the calcinated samples. The XRD patterns show that the crystallite size rises from S1 to S2, and then grows further with calcination temperature from S3 to S4, as shown by the gradual sharpening of diffraction peaks. This suggests that calcination enhances particle growth and crystallinity, likely due to improved atomic arrangement

and reduced imperfections. The values of dislocation density and unit cell volume are shown in Table 1. The variation of FWHM, Lattice parameter, Crystalline size, Strain, and Dislocation density in all four sets of samples is shown in Fig. 1(c).

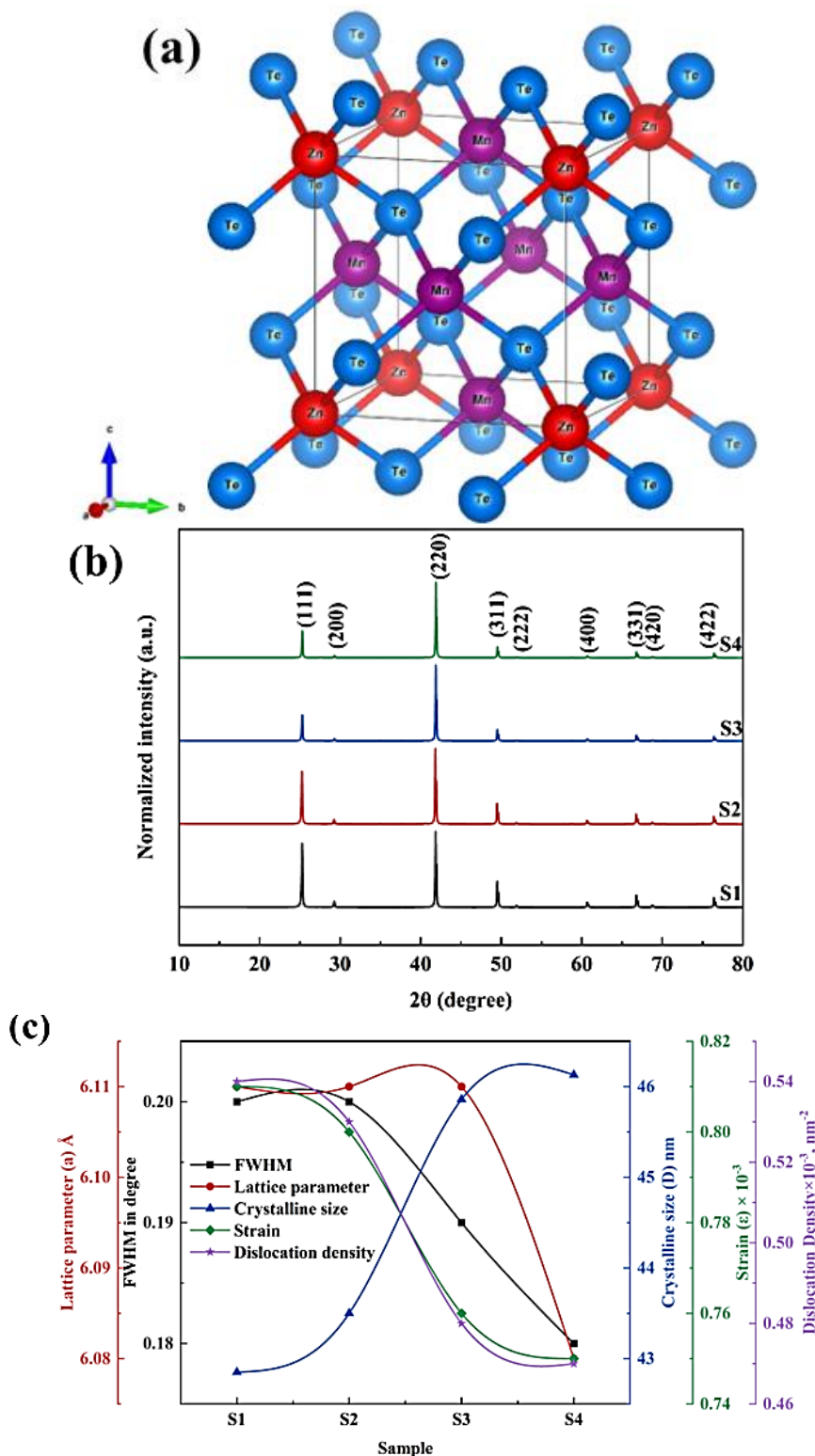


Fig. 1. (a) Crystal Structure of Mn-doped ZnTe, (b) XRD patterns of pure and doped ZnTe NPs, (c) A plot shows the relation in FWHM, Lattice parameter, Crystalline size, Strain, and Dislocation density with samples (colour online)

Table 1. Structural parameters of pure and doped ZnTe samples

Sample	FWHM in degrees	d-spacing	Lattice parameter (a) (Å)	Dislocation density $\times 10^{-3}$ (nm ⁻²)	Unit cell volume (V) (Å ³)
S1	0.20	2.16	6.11	0.54	228.10
S2	0.20	2.16	6.11	0.53	228.10
S3	0.19	2.16	6.11	0.48	228.10
S4	0.18	2.15	6.08	0.47	224.76

3.1.1. Williamson-Hall methods

X-ray diffraction patterns show broadening due to strain from lattice distortions and crystal defects, significantly affected by the diffraction angle, θ . The Williamson-Hall (W-H) method, which relies on a $\tan\theta$ relationship, contrasts with the Scherrer equation's $1/\cos\theta$ dependency. This distinction enables the separation of reflection broadening caused by the combined effects of microstrain and small crystallite size, two key microstructural factors. In the W-H approach, size and strain broadening are treated as additive components of a Bragg peak's total integral breadth, allowing their distinct analysis based on differing θ dependencies [24].

$$\beta_{\text{total}} = \beta_{\text{size}} + \beta_{\text{strain}} \quad (2)$$

In this study, the average crystalline size and microstrain were determined using the W-H equations developed. This eccentric strain essentially influences the physical expansion and the peak expansion of the XRD.

$$\beta_{hkl} \cos\theta = \left(\frac{k\lambda}{D}\right) + \epsilon(4 \sin\theta) \quad (3)$$

Equation 3 is known as the Williamson-Hall equation [25]. The preceding equation is converted to a straight-line equation, which is shown in Fig. 2(a-d), where it is plotted against $4\sin\theta$ (x-axis) vs $\beta\cos\theta$ (y-axis) for the compound. The slope of the straight line determines the internal strain value. The intersection determines the average crystal size of the compound.

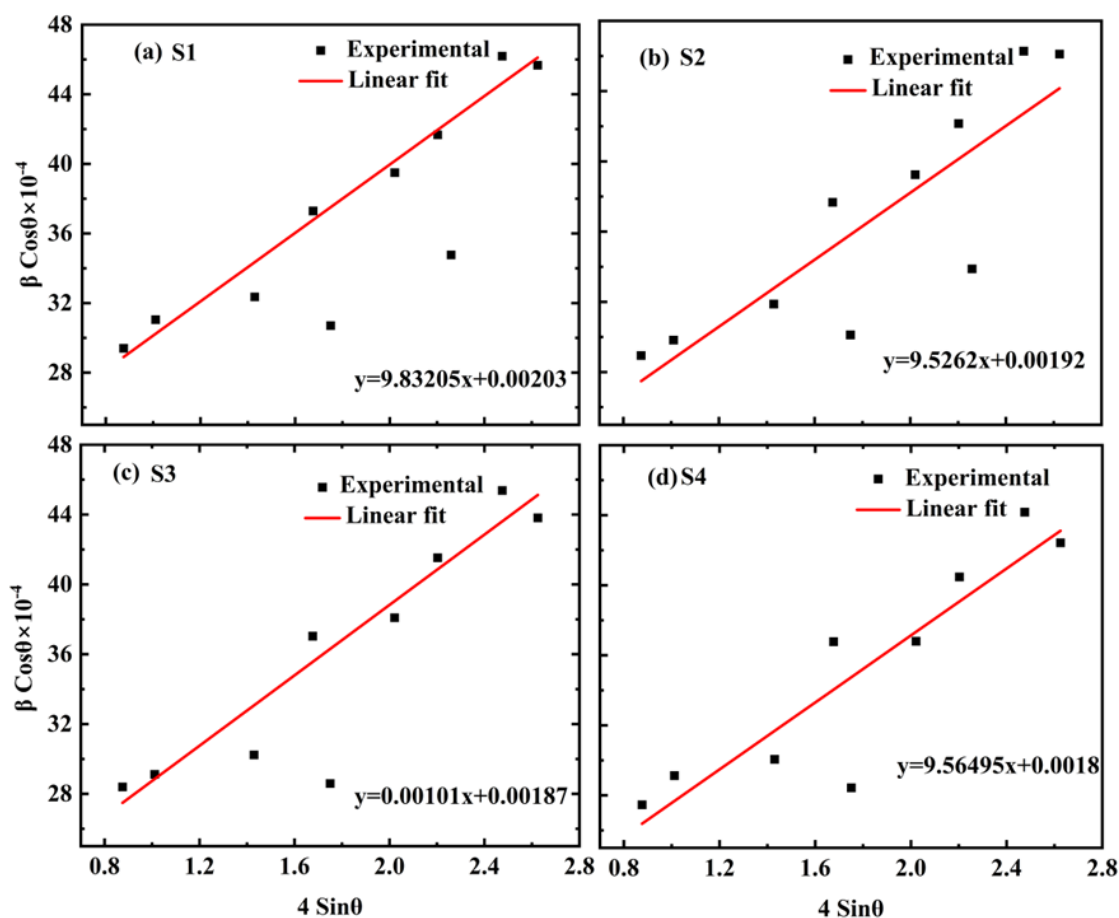


Fig. 2. (a-d) Williamson-Hall plot of pure and Mn-doped ZnTe NPs (colour online)

3.1.2. Modified Scherrer formula (MS)

We employ the XRD technique to investigate nanocrystals, where the sharpness or broadening of diffraction peaks provides insights into the properties of our compound. Broader peaks typically indicate smaller crystal sizes and may also suggest the presence of stress or defects within the crystals. Peak broadening arises from two main sources: physical broadening, related to crystal size and imperfections, and instrumental broadening, inherent to the XRD equipment. We quantify this broadening by measuring the FWHM of the peaks. The crystallite size of ZnTe was determined using the Debye-Scherrer equation (1), i.e. $D = \frac{k\lambda}{\beta_{hkl} \cos \theta}$ based on the X-ray peak broadening. Where k refers to the shape factor=0.9, λ is the wavelength of incident radiation (1.5406 Å), β is the FWHM, and θ is Bragg's diffraction angle.

Using the modified Scherrer equation can reduce potential mathematical errors. By taking the logarithm of

both sides of equation (1), equation (4) is derived. Using the modified Scherrer equation can reduce potential mathematical errors [24]. By taking the logarithm of both sides of equation (1), equation (4) is derived.

$$\ln \beta = \ln \left(\frac{k\lambda}{D} \right) + \ln \left(\frac{1}{\cos \theta} \right) \quad (4)$$

Plotting $\ln(1/\cos \theta)$ versus $\ln(\beta)$ yields a graph where the y-axis intercept gives $\ln(k/D)$. This graph is referred to as the modified Scherrer graph (Fig. 3(a-d)), which enables the calculation of the average crystallite size for samples. The results are summarized in Table 2. By plotting $\ln(1/\cos \theta)$ against $\ln(\beta)$, the slope and intercept of the fitted line can be determined. Using the intercept, the average crystallite size can be calculated with equation (5) based on the following relationship.

$$D = \frac{k\lambda}{e^{\text{intercept}}} \quad (5)$$

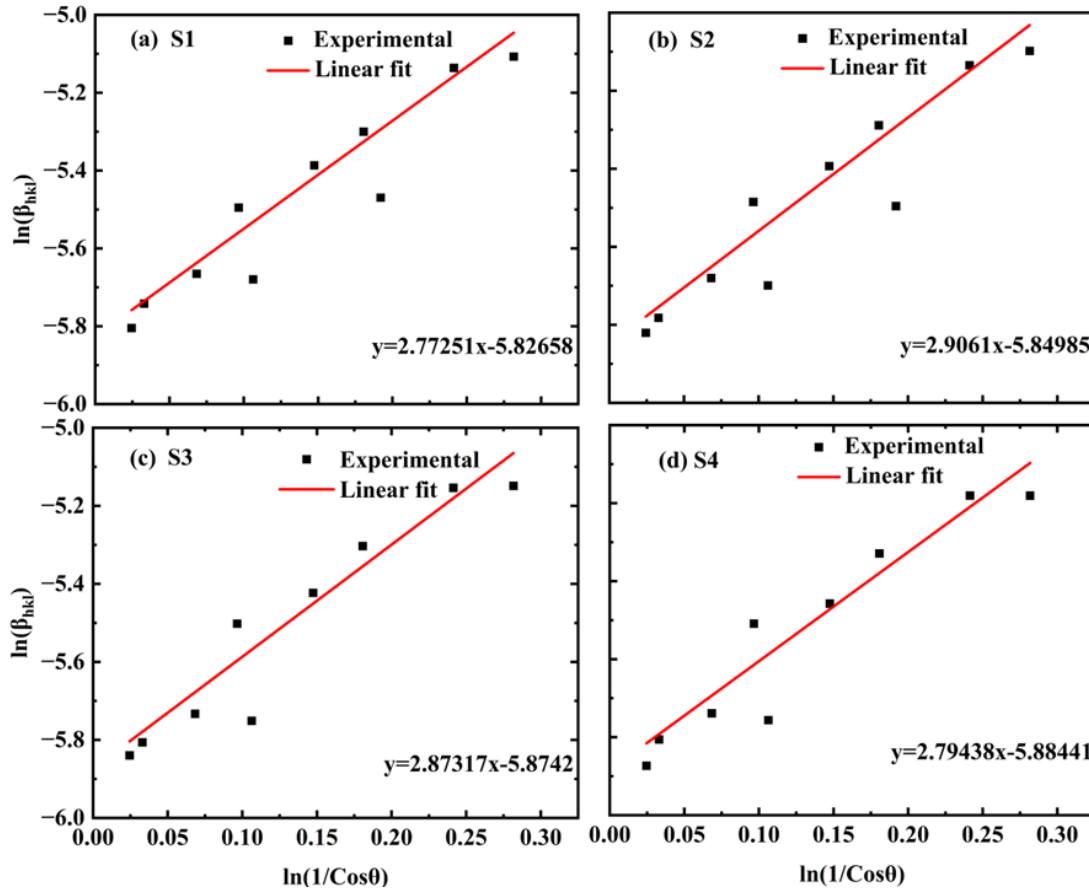


Fig. 3. (a-d) Modified Scherrer method plot for pure and Mn-doped ZnTe samples (colour online)

3.1.3. Halder-Wagner Landford's method (HWL)

The average crystallite size (D) and microstrain (ϵ) were calculated using the Halder-Wagner-Langford (HWL) method, based on the integral breadths (β) derived from XRD data. In the size-strain plot approach, XRD peak broadening is attributed to both crystallite size effects and

lattice strain. However, XRD peaks do not strictly follow a Lorentzian or Gaussian profile. While the Gaussian function closely fits the central peak region, it decays too rapidly at the tails, whereas the Lorentzian function matches the tails well but poorly represents the peak region. To address this discrepancy, the HWL method employs a symmetric Voigt function, which is a convolution of

Gaussian and Lorentzian functions, to model peak broadening. The relationship between crystallite size and lattice strain is then described by the Halder-Wagner equation as follows [26,27]:

$$\left(\frac{\beta^*}{d^*}\right)^2 = \frac{1}{D} \left(\frac{\beta^*}{d^{*2}}\right) + \left(\frac{\epsilon}{2}\right)^2 \quad (6)$$

where, $\beta^* = \frac{\beta \cos \theta}{\lambda}$ and $d^* = \frac{2 \sin \theta}{\lambda}$, λ is the X-ray wavelength (1.5460 Å).

Fig. 4(a-d) shows a plot with $\left(\frac{\beta^*}{d^{*2}}\right)$ on the x-axis and $\left(\frac{\beta^*}{d^*}\right)^2$ on the y-axis. To estimate the crystalline size, use

the formula $D = 1/\text{slope}$, where the slope is obtained from the plot. Similarly, to estimate the microstrain (ϵ), use the formula $\epsilon = (4 \times \text{intercept})^{1/2}$, where the intercept is derived from the plot.

For the precise measurement of crystallite size and microstrain, several line-broadening models were compared. The HWL method gives better accuracy by taking into account actual Voigt peak profiles, whereas the WH approach isolates size and strain effects. Scherrer-based approaches mainly provide estimated crystallite size estimations. The dependability of the acquired microstructural characteristics is confirmed by the consistent trends displayed by all approaches, despite the fact that numerical values vary due to model assumptions.

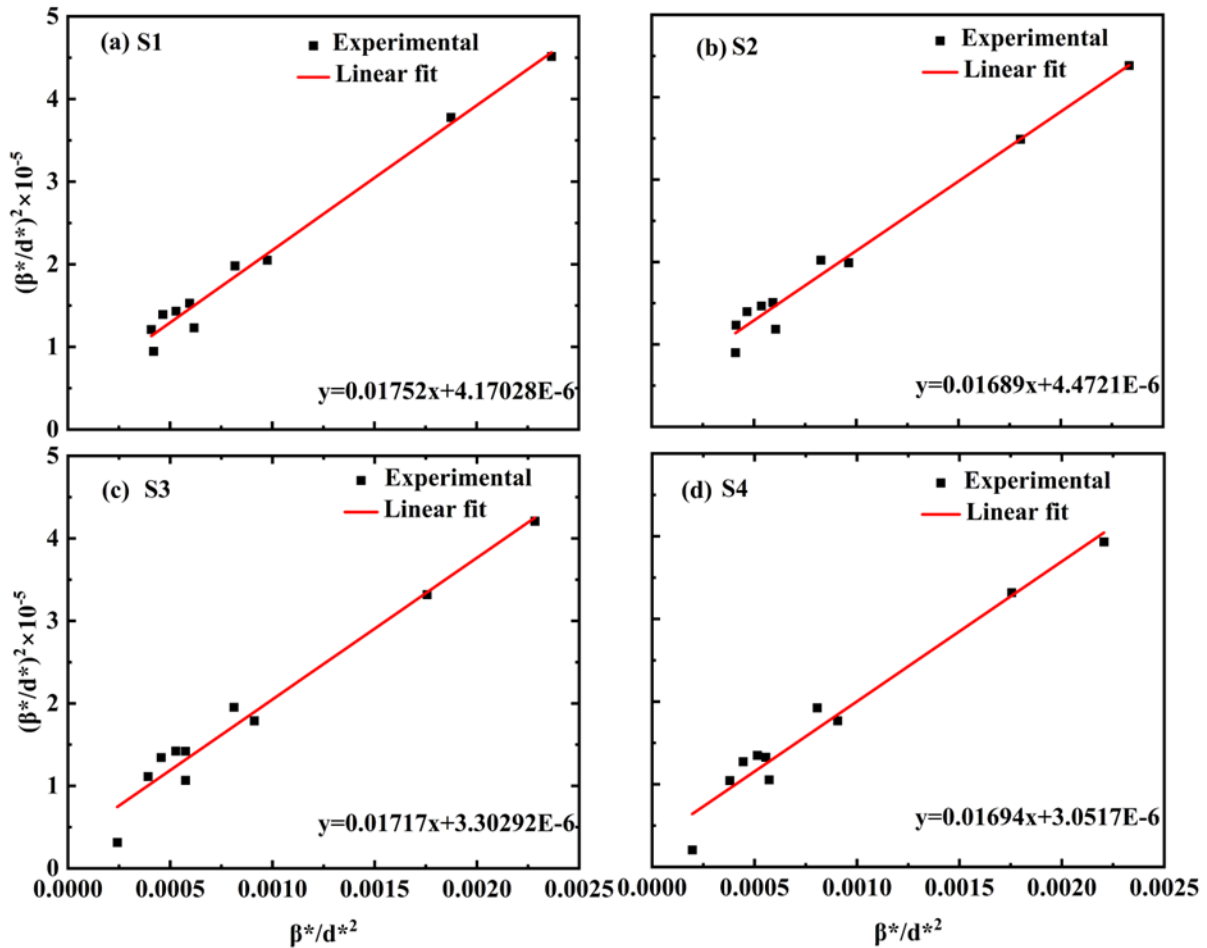


Fig. 4. (a-d) Halder-Wagner Landford's plot of pure and Mn-doped ZnTe NPs (colour online)

Table 2. Variation in the crystalline size and strain in Sherrer method, modified Scherrer, W-H, and HWL methods

Sample	Sherrer Method		Modified Sherrer Method	W-H Method		HWL Method	
	D (nm)	Strain (ϵ) $\times 10^{-3}$	D (nm)	D (nm)	Strain (ϵ)	D (nm)	Strain (ϵ) $\times 10^{-3}$
S1	42.85	0.81	47.03	68.30	9.83	57.08	4.08
S2	43.50	0.80	48.14	73.36	9.53	59.21	4.23
S3	45.86	0.76	49.32	74.14	0.01×10^{-1}	58.24	3.63
S4	46.13	0.75	49.83	77.03	9.56	59.03	3.49

3.2. Morphological study

SEM images of all four sets of samples are shown in Fig. 5(a-d), and Fig. 5(e-h) shows the histogram plot of particle size distribution of pure and doped samples. Fig. 5(a) illustrates the pure ZnTe NPs' irregular grains with smooth surfaces and hexagon-like shapes or uneven shapes. Since undoped ZnTe is produced without further heat treatment, the surface appears rough with some agglomeration. The irregularity points to a polycrystalline structure with little control over grain size. The incorporation of Mn most likely influences the nucleation and growth processes, resulting in a denser structure. Since some agglomeration is still visible, Mn doping by itself does not substantially change the fundamental grain structure in the absence of thermal processing. In comparison to S2, the agglomerated nanoparticle morphology is more refined, with smaller and more homogeneous particles. At 100 °C, calcination probably starts partial sintering, which lowers porosity and improves particle cohesiveness. There is still

some roughness on the surface, indicating that this temperature is not high enough to achieve phase stabilization or full grain growth. Out of the four samples, S4 has the most consistent and distinct particle morphology. The greater calcination temperature (250 °C) improves sintering, resulting in larger, more consolidated grains with lower porosity. The particles seem more faceted, indicating better crystallinity and phase stability as a result of heat energy that facilitates Mn incorporation and lattice rearrangements. When ZnTe is doped with 5 % Mn and calcinated at 100 °C and 250 °C, the average particle size increased from 3.25-40.00 μm compared to undoped ZnTe NPs (Fig. 5(e-h)). Grain size uniformity was further enhanced by raising the temperature, leading to a tighter grain size. These observations align with the XRD results, which indicate an increase in crystallite size [28].

Overall, only slight morphological changes are observed between samples; the principal distinctions are due to the distribution of particles and agglomeration degree, as quantitatively verified by Fig. 5(e-h).

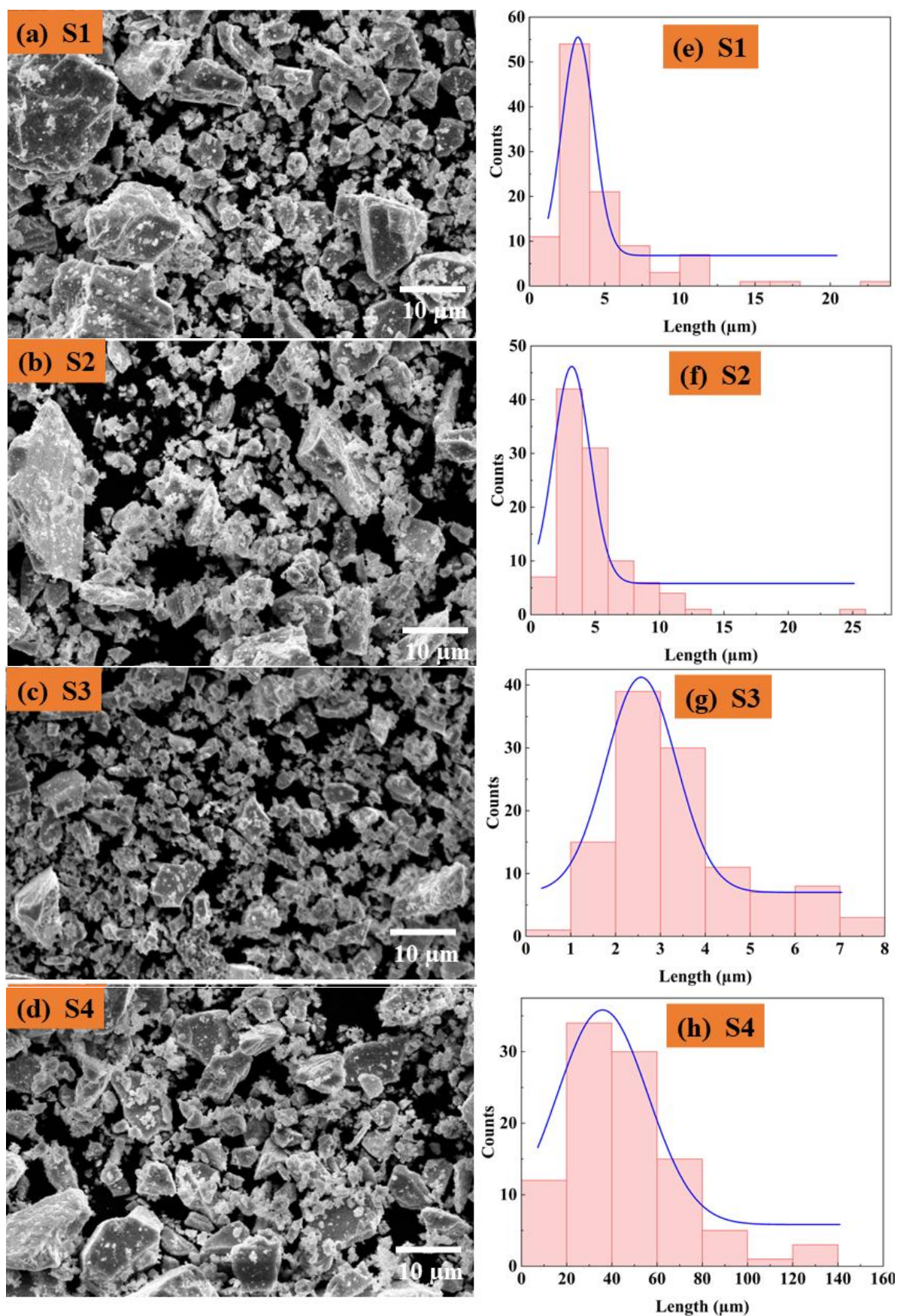


Fig. 5. (a-d) SEM image, (e-h) their corresponding histogram plot of particle size distribution for pure and doped Mn-doped ZnTe NPs (colour online)

3.3. UV-visible and FTIR analysis

UV-Vis Reflectance spectroscopy is a noncorrosive scientific technique to study the optical properties. UV-vis Reflectance spectra were performed on the Mn-doped ZnTe sample to determine its optical band gap, as illustrated in Fig. 6 (a). The band gap energy is the difference in energy between the valence band and the conduction band of a solid material. The optical band gap of the samples was analysed by the Tauc plot [29,30]. Derived from the UV-Vis reflectance spectra. The Tauc's equation is given by:

$$(\alpha h\nu) = A(h\nu - E_g)^n \quad (7)$$

$$(F(R)h\nu) = A(h\nu - E_g)^n \quad (8)$$

$F(R)$ denotes the Kubelka-Munk function [31], which is the ratio of the absorption coefficient (k) to the scattering coefficient (s) in m^{-1} . n can be $1/2$, $3/2$, 2 , or more, depending on whether the transition is direct-allowed, direct-forbidden, indirect-allowed, or indirect-forbidden.

$$F(R) = \frac{k}{s} = \frac{(1-R)^2}{2R} \quad (9)$$

R is the % reflectance obtained in the DRS data.

From Fig. 6(a), it is clear that the absorption wavelength varies according to the change in doping concentration and with varying temperature. The absorption edge shifts towards higher wavelengths with doping. This indicates that the band gap of ZnTe material decreases from 2.11 to 2.06 eV. With the doping concentration up to some critical level, which is known as the Mott critical density.

Above the Mott critical density, the absorption edge shifts towards the lower wavelength region. The lower wavelength shift or increase in the band gap, or blue shift, can be explained by the Burstein-Moss effect [32]. But as we increase the temperature, the band gap again increases from 2.08 to 2.10 eV. The band gap energy of a semiconductor describes the energy needed to excite an electron from the valence band to the conduction band. An accurate determination of the band gap energy is crucial in predicting photophysical and photochemical properties of semiconductors.

The 3d electrons of Mn interact with the host valence and conduction bands via sp-d exchange interactions. Because of the introduction of localized states within the bandgap or a shift in the conduction band edge, this interaction may result in a modest narrowing of the bandgap, which would lower the energy difference after doping of Mn into ZnTe. By calcinating, the sample crystallinity increases and lattice strain decreases, bringing the bandgap closer to its actual value. Some Mn-related defects or disorder are annealed out, limiting the disturbance of the electrical structure, as indicated by the increase of band gap from 2.08-2.10 eV (Fig. (b-e)). Zaari et al. [33] found that the band gap of Mn-doped ZnTe is around 1.49 eV, while Khan et al. [16] observed that the band gap decreases with increasing Mn concentration from 8 % to 16 %. This result is consistent with our findings, where the band gap decreases with doping. By increasing the calcination temperature, crystallinity increases, XRD particle size increases, as confirmed by SEM, and residual defects or Mn clustering decrease. The bandgap approaches 2.10 eV, indicating that thermal energy at this temperature effectively recovers the lattice to a state more similar to pure ZnTe, but not completely, due to residual Mn inclusion.

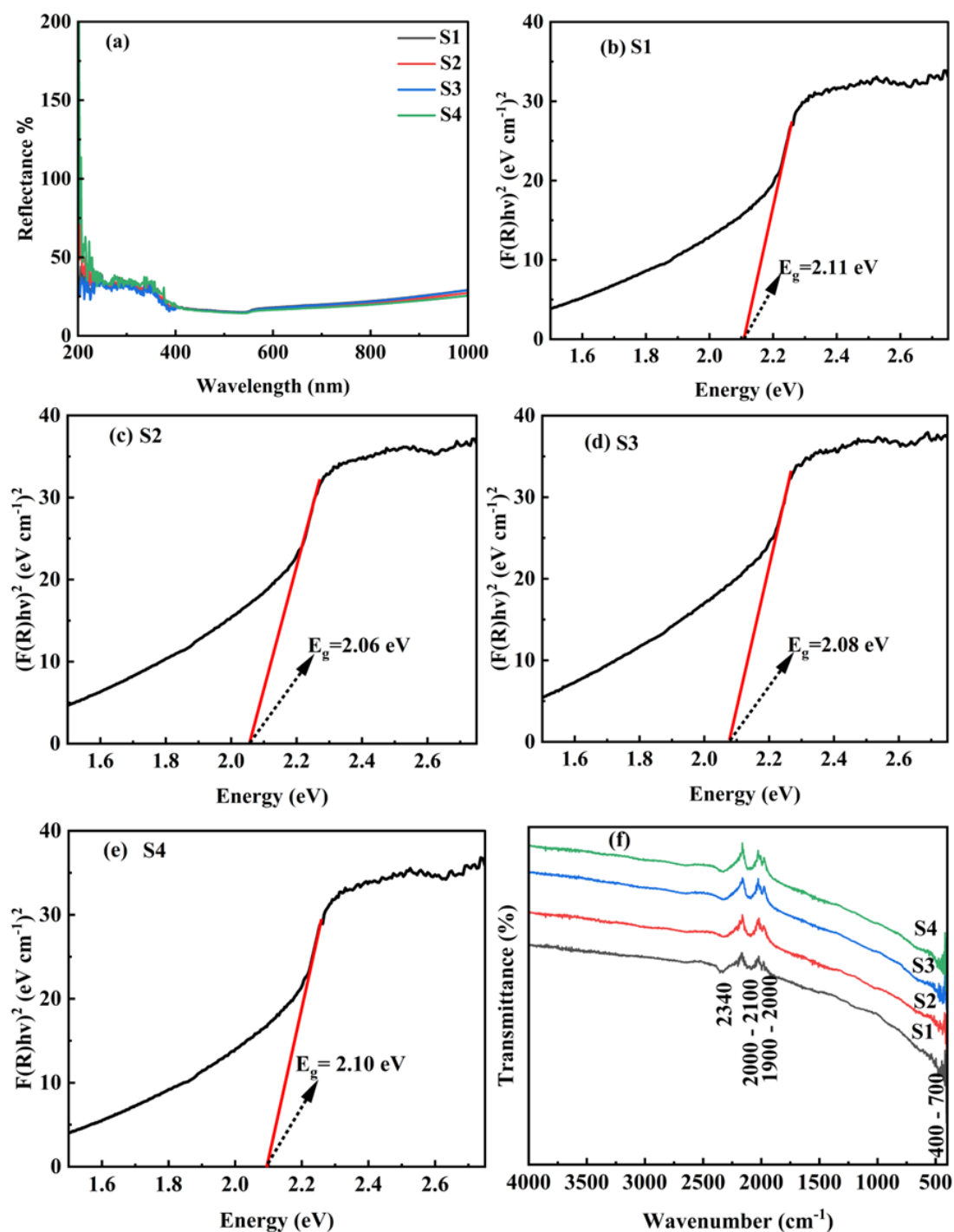


Fig. 6. (a) UV-Visible reflectance spectra, (b-e) direct bandgap of UV-Visible reflectance spectra, (f) FTIR Spectra of pure and Mn-doped ZnTe (colour online)

FTIR spectroscopy is a key technique used to identify chemical bonding and elemental constituents in samples. At room temperature, the FTIR spectra were recorded between 400 and 4000 cm⁻¹ to study the vibrational band located in Mn-doped ZnTe NPs at room temperature, as shown in Fig. 6(f). The peak appears around 2340 cm⁻¹, representing O=C=O stretching, the range between 2100-2000 cm⁻¹ represents C=C or C=N stretching, at 2000 cm⁻¹ it represents C=C=N stretching, and the range between 1900-

2000 cm⁻¹ represents C=C=C stretching. The stretching band of Zn-Te is observed around 400-700 cm⁻¹ in pure and doped samples. There is a very small difference related to the intensity and the peak position. The peaks shift as we move from S1 to S4, corresponding to the successful incorporation of Mn into ZnTe host materials.

4. Conclusions

Pure ZnTe and Mn-doped ZnTe NPs were successfully synthesized using the SSR and characterized through XRD, SEM, FTIR, and UV-Vis techniques. XRD analysis confirmed Mn incorporation into the ZnTe lattice, with an increase in the crystallinity of the sample with increasing temperature. This crystalline size and strain were evaluated using the Scherrer, modified Scherrer, and Halder-Wagner Landford's methods. This confirms that Mn doping increases crystallite size. SEM analysis revealed that average particle sizes increase, aligning well with XRD results. UV-Visible analysis confirmed that Mn doping effectively reduces the band gap from 2.11 to 2.06 eV, while increasing the calcination temperature leads to a slight widening of the band gap. This tunability demonstrates that Mn incorporation and thermal treatment play a crucial role in tailoring the electronic structure of ZnTe. The ability to adjust the band gap according to processing conditions enhances the suitability of Mn-doped ZnTe nanoparticles for visible-light-driven photocatalysis and optoelectronic device applications. Overall, the findings highlight the potential of controlled doping and calcination as effective strategies for engineering the functional properties of ZnTe-based nanomaterials.

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Data availability

The data that support the findings of this study are available from the corresponding author upon reasonable request.

Declaration of competing interest

The authors declare that they have no known financial/interpersonal conflicts that might be perceived as having impacted the research disclosed in this paper.

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*Corresponding author: virendra.verma@vitap.ac.in