Effect of dopant concentration Mn in SnO₂ nanoparticles on photocatalytic, magnetic and optical properties

R. Renuga^{a,*}, S. Srinivasan^a, T. Thangeeswari^b, S. Suresh^c, E.Priyadharshini^d, R. Bomila^e

^aDepartment of Physics, Presidency College, Chennai – 600 005, India ^bDepartment of Physics, Vel Tech Multitech Engineering College, Chennai-600 062, Tamilnadu, India ^cDepartment of Physics, Saveetha Engineering College (Autonomous),

Thandalam, Chennai -602105, Tamil Nadu, India

^dDepartment of Physics, Jaya Engineering College, Thiruninravur, Tamil Nadu, India

^eDepartment of Physics, Valliammai Women's College, Thirkovilur, Tamil Nadu, India

Mn doped SnO₂ nanoparticles were synthesized in a sol-gel technique at various concentrations. A wide range of properties are analyzed by using X-ray diffraction (XRD), Fourier transform infrared spectroscopy (FTIR), scanning electron microscopy with energy dispersive spectroscopy (SEM with EDAX), Transmission Electron Microscopy (TEM), UV-Visible spectroscopy, photoluminescence spectroscopy (PL), Vibrating Sample Magnetometer (VSM), and photocatalytic studies. Results from EDAX confirmed the presence of Sn and Mn ions in SnO₂ nanoparticles doped with Mn. As a result of analyzing the luminescence of all synthesized samples, blue emission characteristic of lighting applications was observed. Furthermore, 0.2M Mn doped SnO₂ sample shows the enhanced ferromagnetism at room temperature with elevated saturation magnetization value. The 0.2M Mn doped SnO₂ nanoparticles show the significant increase in photocatalytic efficiency of SnO₂ among the other doped samples. Thus, all experiment results demonstrated that the concentration of dopant influences the structure, optical properties, photocatalytic properties, and magnetic properties of SnO₂.

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1. Introduction

The analysis of structural and optical properties of nanostructured transition metal oxides has a deep-seated function in the understanding and advancement in the requirement of electronic devices [1,2]. As a result of its physical and chemical interactions with adsorbed species, as well as its capability to function at low temperatures, tin oxide is a very capable and efficient material [3–5]. It has distinctive properties such as chemically inert, mechanically hard and thermally heat resistant. Tin oxide (SnO₂) is n-type wide bandgap semiconductor (Eg = 3.6 eV at 300 K) which is used in various electronic instruments, such as gas sensors, solar cells, liquid crystal displays and photovoltaic cells[6–8]. It is also important to consider whether a material is crystalline or amorphous in terms of its physical and chemical properties. SnO2 nanoparticles can be synthesized in several different sizes, which enhance their electrical, morphological, magnetic, and optical properties [9]. The synthesis process determines the chemical and physical properties of tin-oxide, such as particle size, shape, and crystalline. Nano-sized SnO₂ was prepared using various techniques such as sol-gel [10,11], chemical co-precipitation [12], sonochemical process [13]and other methods. Nevertheless, the sol-gel technique has a number of advantages over the

^{*} Corresponding author: vrevanth11@gmail.com https://doi.org/10.15251/DJNB.2022.174.1475

other available synthesis techniques for the production of tin oxide. Synthesize method has a lower processing temperature, improved homogeneity, and controlled stoichiometry, as well as a low cost [14]. By doping various transition metals have improved the properties in tin oxide nanomaterials such as enriched sensitivity, tremendous selectivity, rapid response and recovery behavior [6]. New studies have shown that transition metal doping with tin oxide resulted in room-temperature ferromagnetism, as well as significant changes in sample optical properties [4-5].

Photocatalysis is a safe and effective way to remove dyes from the textile dye industry wastewater. Research findings showed that the doping of transition metals in semiconductor metal oxide nanoparticles increases the photo catalytic activity in industry chemical waste [15-17]. Tin oxide dilute magnetic semiconductors (DMS) have received a lot of attention due to their use in spintronic applications. It is observed that the ferromagnetism at room temperature is achieved by doping transition metals with tin oxide [18]. Manganese doped SnO₂ shows room temperature ferromagnetism with high saturation magnetization. Further, Mn doped SnO₂ material has been shown to be a promising material for white light emitting diodes (LED) [19]. Divalent Mn²⁺ ions have excellent luminescent properties due to their broad emission between the regions from green colour to red when irradiate by ultraviolet (UV) excitation. Mn²⁺ has the demand for white light emitting diodes [20].

In this study, we have investigated the physical, morphological, optic, and magnetic properties of SnO_2 nanoparticles doped with transition metal manganese. Manganese is one of the preferred dopants because the manganese particles can be fluently substituted inside the SnO_2 lattice. Mn atoms were embedded into the SnO_2 lattice by altering the Mn concentrations, and the effect of doping was well-proven.

2. Experimental procedure

2.1. Material synthesis

Tin chloride (SnCl₂.H₂O) and manganese chloride (MnCl₂.4H₂O) were employed as metal precursors. The concentrations of 0.1 M of Mn, and 0.9 M of SnO₂ were dissolved in 50 ml of double- distilled water. 15 ml of aqueous ammonia (3M) is added drop wise to precursors under stirring for 2h. After fully adding the ammonia solution, the admixture becomes a gel. The product is heated to dematerialize the water molecule and also dried in a hot air oven for about 4 hours at 80°C. Then finally the product was calcinated at 400°C in muffle furnace for 2h. Same route, 0.2M and 0.3M Mn doped SnO₂ nanoparticles were prepared. For comparison pure SnO₂ nanoparticle also synthesized by above mentioned method. Samples are named as pure, 0.1, 0.2, 0.3 Mn doped SnO₂ nanoparticles.

2.2. Photocatalytic testing

The degradation of methylene blue (MB) dye in aqueous solution under visible light irradiation of pure SnO2, 0.1, 0.2 and 0.3 Mn doped SnO₂ nanoparticles was analyzed. Samples of 5 mg of pure SnO₂, 0.1, 0.2 and 0.3 Mn doped SnO₂ are added to 5 mg/L of MB dye. The solution is stirred for 30 minutes and stored in a dark place to reach adsorption-desorption equilibrium before the interaction of visible light. The initial concentration is represented by (C₀). The mixed solution was used for 5 ml of the solution and the visible spectra of the MB solution were monitored every 30 minutes. The percentage of dye degradation is predicted by

$$\eta = [(C_0 - C_t) / C_0] \times 100$$

where η is the percentage of degradation, C₀ is the concentration of the dye (mg/L) at time 0 min and Ct is the concentration of the dye after irradiation at a time interval of 30 min.

3. Results and discussion

3.1. X-Ray diffraction studies

XRD patterns of pure and SnO₂ nanoparticles doped with 0.1, 0.2 and 0.3 Mn is depicts in figure 1. The diffraction peak is at different angles for all samples such as 26.71°, 33.94°, 38.28°, 51.92°, 54.82°, 58.36°, 61.89°, 64.94°, 66.22°, 71.21°, and 78.91° corresponds to (110), (101), (200), (211), (220), (002), (310), (112), (301), (202), and (321) lattice planes [12-21]. All diffraction peaks of tin oxide nanoparticles with 0.1, 0.2 and 0.3 Mn are the same as pure SnO₂ nanoparticles. For 0.2, and 0.3 Mn, we observed impurity peaks at angles of 23.05° and 32.66° corresponding to Mn₂O₃ [22-23]. Therefore, XRD studies confirm that the tetragonal structure of SnO₂ and the Mn doping also does not introduce any change in the tetragonal structure of SnO₂ nanoparticles. The decrease in peak intensity of 0.1, 0.2 and 0.3 Mn-doped SnO₂ nanoparticles can also be attributed to the replacement of Sn⁴⁺ ions by Mn²⁺ ions in the SnO₂ lattice [24]. Also the diffraction peaks are shifted to smaller angles due to the Mn doping.



Fig. 1. XRD pattern of Pure, 0.1,0.2, and 0.3 Mn doped SnO₂ nanoparticles.

Using the full-width half-value of the high-intensity diffraction peak (111), the average crystal size is estimated via the Scherrer formula:

$D = 0.89\lambda/\beta \cos\theta$

where D represents the size of the crystallite particle, λ represents the wavelength of the incident X-rays, β represents the full width at half maximum, and θ represents the Bragg diffraction angle. Pure SnO2 particle size, 0.1, 0.2, 0.3 Mn-doped SnO₂ particle size are 47.59 nm, 14.21 nm or 29.25 nm, 26.69 nm. The particle size of SnO₂ nanoparticles doped with 0.1, 0.2, and 0.3 Mn decrease compared to pure SnO₂ nanoparticles. Incorporation of divalent Mn²⁺ ions into Sn⁴⁺ ions creates the oxygen vacancy configuration that leads to particle size reduction [24]. Further, particle size of 0.2, 0.3 Mn doped SnO₂ nanoparticles is increased compared to 0.1 Mn doped nanoparticles which may correspond to impurity Mn₂O₃ phase.

3.2. FTIR spectra analysis

FTIR spectroscopy is used to study chemical bonds present in the synthesized samples. FTIR spectra of pure, 0.1, 0.2 and 0.3 Mn-doped SnO_2 nanoparticles are shown in figure 2. The broad peak at 3440 cm⁻¹ for all synthesized samples represents the characteristics of O-H

stretching vibration. The sharp peak at 510 cm⁻¹ for all samples such as pure, 0.1, 0.2, and 0.3 Mndoped SnO_2 nanoparticles corresponds to Sn-O vibration and the peaks of Mn doped samples shifted to higher wavenumber which may the characteristics of reduction in particle size [25-26]. Twisting mode of OH groups is identified by another weak peak at 1633 cm⁻¹.



Fig. 2. FTIR Spectra of Pure, 0.1, 0.2, and 0.3 Mn doped SnO₂.

3.3. Morphological study

Figure 3 (a-d) shows the SEM images of pure, 0.1, 0.2, and 0.3 Mn-doped SnO_2 nanoparticles. It is an evidence for the indeterminate agglomerated morphology of SnO_2 nanoparticles. For increase in concentration of Mn, more particles are distributed on its surfaces which can affect the optical properties of synthesized samples. Especially in 0.2 Mn-doped SnO_2 nanoparticles, it is noticed that the particle distribution is more on its surfaces.



Fig. 3. (a-d). Scanning electron micrographs of (a) pure (b), 0.1 Mn SnO2 (c), 0.2 Mn SnO₂ (d) 0.3 Mn SnO₂

EDAX is used to identify the percentage of doping and purity of the particular sample. The EDAX spectra of pure, 0.1, 0.2 and 0.3 $Mn:SnO_2$ are revealed in figure 4(a-d). EDAX results confirmed our precursor composition which we mentioned in the synthesized process. The obtained atomic concentration of the elements with respect to pure, 0.1, 0.2, and 0.3 Mn-doped SnO_2 nanoparticles was mentioned in table 1. Hence the existence of Mn in SnO_2 nanoparticles is confirmed by structural and elemental characterization.

| Particle Name | Atomic % | Atomic % | Atomic % |
|-------------------------|----------|----------|----------|
| | of Sn | of Mn | of O |
| SnO ₂ | 36.18 | | 56.65 |
| 0.1 Mn SnO ₂ | 34.78 | 9.45 | 55.77 |
| 0.2 Mn SnO ₂ | 15.09 | 18.57 | 66.34 |
| 0.3 Mn SnO ₂ | 9.74 | 30.23 | 60.01 |

Table 1. Atomic concentration of elements for pure SnO₂, 0.1, 0.2 and 0.3 Mn-doped SnO₂ nanoparticles *from the EDAX analysis.*

Figure 5(a-d) shows the TEM images of the un-doped and Mn-incorporated SnO_2 nanoparticles. The result confirms that the well changes take place in structural properties due to the incorporation of Mn in SnO_2 lattice. Further it confirms the existence of single crystalline nature which is concordance with our XRD results.

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Fig. 4. (a-d) EDAX Spectra of (a) pure SnO₂ (b), 0.1 Mn SnO₂ (c), 0.2 Mn SnO₂ (d) 0.3 Mn SnO₂.



Fig. 5 (a-d). TEM of (a) pure SnO₂ (b), 0.1 Mn SnO₂ (c), 0.2 Mn SnO₂ (d) 0.3 Mn SnO₂.

3.4. Optical studies

The UV- visible absorption non destructive method is used to observe the changes in electronic configuration of samples and thereby identify the enhanced optical properties. The UV-visible absorption spectra in the wavelength between 200 to 800 nm for pure, 0.1, 0.2 and 0.3 Mn-doped SnO₂ nanoparticles are shown in figure 6. As a result of absorption, the absorbance of pure, 0.1, 0.2, and 0.3 Mn-doped SnO₂ nanoparticles shows the peak around 298 nm. This peak corresponds to the process of photo- excitation of charge carrier electrons from valence band to conduction band [5]. It is noticed that the considerable higher shift in absorbance spectra of 0.1, 0.2, and 0.3 Mn-doped SnO₂ nanoparticles. The absorbance depends on various factors such as addition of impurity, oxygen vacancy, and bandgap.

The optical band gap (Eg) of a semiconductor is connected to the optical absorption coefficient (α) and the incident photon energy (hv) by Mott and Davis relation

$$\alpha hv \sim (hv - E_g)^{1/2}$$

By linear extrapolation of the curves to $(\alpha hv)^2$ and photon energy hv, energy gap value is determined. From figure 7 (a-d), it was obtained that the band gap values of pure SnO₂, 0.1, 0.2 and 0.3 Mn-doped SnO₂ nanoparticles are 3.29, 3.10, 3.19 and 3.20 eV respectively. It confirms the band gap value of 0.1, 0.2 and 0.3 Mn-doped SnO₂ nanoparticles found to be lesser than the band gap value of pure SnO₂. The invention of a new dopant energy level of Mn ions in SnO₂ corresponds to reduction in bandgap of 0.1, 0.2 and 0.3 Mn-doped SnO₂ nanoparticles.



Fig. 6. UV-Vis absorption spectra of pure, 0.1, 0.2, 0.3 Mn SnO₂ Nanoparticles.

This reduction in bandgap confirms the presence of Mn ions at the SnO_2 lattice and changes the optical properties by tuning the bandgap energy [4].



*Fig. 7. (a-d). Band gap spectra of (a) pure (b), 0.1 Mn SnO*₂ *(c), 0.2 Mn SnO*₂ *(d) 0.3 Mn SnO*₂ *Nanoparticles.*

3.5. Photoluminescence studies

Figure 8 illustrates the photoluminescence spectra of pure SnO₂, 0.1, 0.2 and 0.3 Mndoped SnO_2 nanoparticles from 300 to 600 nm. For the samples of pure SnO_2 and 0.1 Mn-doped SnO_2 nanoparticles, the intense peak at 329 nm represents the direct near band edge emission (NBE) [10]. It is observed that the peak intensity for 0.2 and 0.3 Mn-doped SnO_2 nanoparticles are decreased drastically when compared to pure SnO₂, and 0.1 Mn-doped SnO₂ nanoparticles. This reduction in peak intensity may be attributed to presence of defects and oxygen vacancies which is consistent with our XRD results [13]. Also for 0.2 and 0.3 Mn-doped SnO₂ nanoparticles, the peak in the UV region shifted to higher wavelength. This red shift in wavelength for 0.2 and 0.3 Mndoped SnO_2 nanoparticles may be due to more concentrations of Mn or presence of impurity Mn_2O_3 [24]. Other hand, the red shift in the UV region may be attributed to reduction in band gap. Further, it is observed that the broad visible luminescence between 400-600 nm which confirms that the optical emission arises from both SnO_2 and dopant Mn. The supplementary peaks around 360-393 nm were observed for 0.2 and 0.3 Mn-doped SnO₂ nanoparticles which may correspond to the existence of defects like oxygen vacancies inside the SnO_2 nanoparticles [26]. Hence, from these results we can conclude that the optical properties of synthesized samples depend on various factors such as concentration of dopant Mn, size of particle and its morphology.



Fig. 8. PL spectra of pure SnO₂, 0.1 Mn SnO₂, 0.2 Mn SnO₂, 0.3 Mn SnO₂ Nanoparticles.

In general, the combination of blue, yellow and green light gives white light as output. Color coordinates of synthesized samples were analyzed by using the CIE chromaticity diagram. CIE chromaticity diagram of pure SnO_2 , 0.1, 0.2 and 0.3 Mn-doped SnO_2 nanoparticles is shown in figure 9(a-b). The chromaticity coordinate values of pure SnO_2 , 0.1, 0.2 and 0.3 Mn-doped SnO_2 nanoparticles is given in table.2. From this experimental analysis, it is noticed that color coordinates of both pure SnO_2 , and Mn doped SnO_2 appeared in a strong blue region which coincides with our blue emission in PL analysis [27].



Fig. 9(a-b). CIE Chromaticity diagram of (a) Pure, 0.1 Mn SnO₂, (b) 0.2, and 0.3 Mn SnO₂.

Table 2. Chromaticity coordinates values of pure SnO₂, 0.1, 0.2 and 0.3 Mn SnO₂ nanoparticles.

| Nanoparticles | CIE Coordinates | | |
|----------------------|-----------------|-------|--|
| | Х | у | |
| SnO ₂ | 0.125 | 0.125 | |
| 0.1 SnO ₂ | 0.106 | 0.183 | |
| 0.2 SnO ₂ | 0.232 | 0.271 | |
| 0.3 SnO ₂ | 0.127 | 0.066 | |

The photoluminescence studies confirm that the prepared nanoparticles are potential resources for blue light emitting diode lamps and displays.

3.6. Magnetic studies

Tin dioxide semiconductor is considered a more appropriate material for the design of dilute magnetic semiconductors. Since it possess the good optical transparency, local oxygen vacancies and more charge carrier density. Figure 10 shows the M-H curves of pure SnO₂, 0.1, 0.2 and 0.3 Mn-doped SnO_2 nanoparticles. For all synthesized samples, at the low magnetic field, we observed the hysteresis loop and hence it possesses the ferromagnetic property at room temperature. The existence of ferromagnetism in pure SnO_2 may arise from the oxygen vacancies which formed during synthesis process [21]. Figure 10 shows that the saturation magnetization decreases for 0.1 and 0.3 Mn-doped SnO₂, but increases for 0.2 Mn-doped SnO₂ nanoparticles. This may correspond to arrest of motion of electrons in the reduced particle size which leads to decrease in magnetic moments [21]. Furthermore, the coercivity value is low for 0.1 and 0.3 Mndoped SnO₂ nanoparticles, which confirm that the ferromagnetism is reduced and changes towards the super paramagnetic nature. But in contrast, coercivity value is high for 0.2 Mn-doped SnO_2 nanoparticles, which may be attributes to more oxygen vacancies from Mn₂O₃ phase. This result is well matched with our EDAX reports also. From these results we can conclude that the enhancement in ferromagnetism depends on dopant concentration which gives more oxygen vacancies. The observed magnetic parameters are shown in table 3.



Fig. 10. M-H curves of pure SnO₂, 0.1, 0.2 and 0.3 Mn-doped SnO₂.

| Compound Name | Saturation Magnetization (emu/g) | Coercivity (Oe) | Retentivity (emu/g) |
|-------------------------|--|-----------------|-------------------------|
| SnO ₂ | 6.132 ×10 ⁻³ | 400.59 | 1.852 ×10 ⁻³ |
| 0.1 Mn SnO ₂ | 2.824×10 ⁻³ | 227.81 | 432.93×10 ⁻⁶ |
| 0.2 Mn SnO ₂ | 8.7×10 ⁻⁶ | 391.82 | 2.07×10 ⁻³ |
| 0.3 Mn SnO ₂ | 3.522×10 ⁻³ | 213.45 | 416.86×10 ⁻⁶ |

Table 3. Magnetic constraints of Pure, 0.1, 0.2 and 0.3 Mn-doped SnO₂.

3.7. Photocatalytic activities

In recent years, due to the wide population of textile industries, the water is contaminated and polluted by its affected echo system. The metal oxide nanoparticle acts as a catalyst for degrading the textile dyes like MB, congo red, and Rhodamine B etc since it has enhanced antibacterial activity. By the process of degradation of methylene blue dye under UV radiation wavelength at 637 nm, photocatalytic activity of Mn doped SnO_2 nanoparticles are analyzed. Under dark conditions there is no adsorption that takes place in the synthesized samples. Photocatalytic activity measured at different interval of time such as 30 min, 60 min, 90 min, 120 min and 150 min adsorption and desorption takes place in photocatalytic material and MB dyes.



Fig. 11(a-d) Time dependent absorbance spectra of pure SnO_2 , 0.1, 0.2 and 0.3 Mn-doped SnO_2 .

Figure 11 (a-d) shows the characteristics of absorbance for pure SnO_2 , 0.1, 0.2 and 0.3 Mn-doped SnO₂ nanoparticles, on MB dye at regular time intervals. It was noticed that for all three synthesized samples, the absorbance reduced as the irradiation time increased. Hence MB dye color changes from dark to light which means it gets degraded [17]. In depth, after 150 min of irradiation under UV radiation, the degradation percentage is achieved 80%, 70%, 55% and 35% respectively for 0.2 M Mn, 0.3 M of Mn, 0.1 M of Mn and pure SnO₂ samples. The 0.2 M of Mn shows the high photocatalytic efficiency when compared to other samples. The factors responsible for this enhancement in 0.2 M of Mn are reduction in crystallite size, oxygen vacancy and larger surface area. In general, larger surface area of the sample enriches the diffuse of reactant molecules to the active site. Further, it resulted in an enhanced loading of the dye molecules and ended with excellent photodegradation. The effect of the synthesized nanoparticles on the absorbance change of MB dye at various time intervals is shown in figure 12(a) and the relation between Photodegradation rates of MB dye with pure SnO₂, 0.1, 0.2 and 0.3 Mn-doped SnO₂ nanoparticles is shown in figure 12(b). This attitude indicates that the photodegradation efficiency improved with increasing dopant Mn concentration, but for 0.2 Mn doped SnO₂ shows more efficiency.

Figure 12 (c) reveals the pseudo-first order kinetics of the MB degradation of the pure and Mn doped SnO_2 . Degradation efficiency estimated by the equation

 $\ln \left(C_0 / C_t \right) = Kt$

K is the pseudo-first-order cost constant, while C_0 and C_t , respectively, are the dye concentrations at time zero and t. The catalytic SnO₂ nanoparticles produced electron-hole pairs after UV treatment. Both the holes and the electrons that were originally intended for the valence band are still there. The hole combines with the water molecules in this process to produce highly reactive non-hydroxyl radicals. Additionally, oxygen peroxide radical anions are created when photo generated electrons inside the conduction band interact with O₂ (O²⁻). Additionally, the highly reactive hydroxyl and superoxide radicals produced cause the methylene blue dye to degrade.

$$SnO_{2} + hv \longrightarrow SnO_{2} (e^{-}_{CB} + h^{+}_{VB})$$
$$SnO_{2} (e^{-}_{CB}) + O_{2} \longrightarrow SnO_{2} + O_{2}^{-}$$
$$SnO_{2} (h^{+}_{VB}) + H_{2}O \longrightarrow OH^{-} + H^{+}$$

 $O_2^- / OH^- + dye (MB) \rightarrow oxidative decomposition \rightarrow CO_2 + H_2O$



Fig. 12. (a). Degradation curves of pure SnO₂, 0.1, 0.2 and 0.3 Mn-doped SnO₂.



Fig. 12. (b) Degradation efficiency curves of pure SnO₂, 0.1, 0.2 and 0.3 Mn-doped SnO₂.



Fig. 12. (c) Kinetic curve of degradation of MB dye in pure SnO₂, 0.1, 0.2 and 0.3 Mn-doped SnO₂.

4. Conclusion

We synthesized pure SnO_2 , SnO_2 nanoparticles doped with 0.1, 0.2 and 0.3 Mn. Various characterization methods such as XRD, FTIR, SEM, TEM, UV, PL, CIE, VSM and photocatalytic activity were used to study the effect of Mn doping concentration on SnO_2 nanoparticles. After doping Mn with SnO_2 , the particle size and band gap value decrease. Magnetic measurements show that the saturation magnetization for the 0.2 Mn-doped SnO_2 nanoparticles increases due to the arrest of electron motion with the reduced particle size. From all characterization analysis it is concluded that of all synthesized nanoparticles, 0.2Mn-doped SnO_2 nanoparticles show enhanced optical, magnetic, and photocatalytic activities. In addition, its use in blue light emitting diode applications can be explored. SnO_2 nanoparticles can be used as cheap, thermally stable and more efficient wastewater management due to its enrichment with photocatalytic activity. Finally, from these results, we can conclude that we can tune the optical, magnetic, and photocatalytic properties of SnO_2 nanoparticles by choosing the appropriate dopant and its concentration, thus bringing them into play in various optoelectronic devices.

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