**Research Article** 

# Green synthesis of SnO<sub>2</sub> nanoparticles using *Delonix elata* leaf extract: Evaluation of its structural, optical, morphological and photocatalytic properties



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Received: 11 June 2020 / Accepted: 16 September 2020 / Published online: 25 September 2020 © Springer Nature Switzerland AG 2020

#### Abstract

This research article reports the green synthesis of SnO<sub>2</sub> nanoparticles and its structural, optical, morphological and photocatalytic behaviors has been achieved by using *Delonix elata* leaf extract. Three different synthesis routes (i.e., sonication, wet chemical, and microwave method) are employed to prepare SnO<sub>2</sub> nanoparticles. A comparative analysis was performed on the main physical properties to get a broader understanding of the effect of processing parameters on prepared SnO<sub>2</sub> nanoparticles. The prepared SnO<sub>2</sub> nanoparticles were characterized using different techniques like TG, XRD, FESEM, EDX, FT-IR, UV–Vis, PSD, and BET surface area analysis. Furthermore, the application of the prepared SnO<sub>2</sub> nanoparticles as photocatalyst was proceeded to degrade rhodamine B dye.

Keywords Green synthesis · SnO<sub>2</sub> NPs · Delonix elata leaf extract · Photocatalytic applications

# **1** Introduction

In the Present-day society, industrial wastewater with an extreme level of organic dyes has raised with the ratio of industrial development. These organic dyes from the outlet of various manufacturing sectors such as plastic, textile, and leather, food, pharmaceutical, and chemical are hazardous to humans [1]. Apart from various diseases and disorders caused by this high toxic of organic dyes, it also severely affect the ecosystem and cause dangerous pollutions (i.e.,) groundwater contamination [1]. The photosynthetic process was limited in the aquatic plants by blocking sunlight, and the oxygen capacity of water was reduced for the normal growth of marine animals due to the high toxicity of organic dyes [2]. Removal of dangerous dyes and toxic organic compounds from wastewaters,

photocatalysts process is the most commonly employed techniques [3]. In the past few decades, enormous effects have been taken for health risk assessment on dangerous dyes and against pollutants to reduce groundwater contamination. Wide range of photocatalysts employing nano metal oxides like titanium dioxide  $(TiO_2)$ , zinc oxide (ZnO), tin oxide (SnO<sub>2</sub>), cadmium sulfide (CdS) and etc., [4–7]. It has proved its properties in degrading a heterogeneous of distinct organic dyes and other pollutants of water into degradable compounds. Amid the dissimilar types of photoactive catalysts, nano metal oxides have been usually premeditated for this reason [8-10]. Various dopants are also used to enhance the dye degradation properties of the aforementioned nano photocatalyst [11-13]. One of the best nano photocatalysts in nano metal oxides with an excellent n-type semiconductor having bandgap energy

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SN Applied Sciences (2020) 2:1735 | https://doi.org/10.1007/s42452-020-03534-z

of 3.6 eV was tin oxide  $(SnO_2)$  nanoparticles [14]. Tetragonal crystal configuration of  $SnO_2$  nanoparticles has latent applications in the pitch of catalysis, rechargeable lithium batteries, optoelectronic devices, and transparent conducting electrodes [15–18].

Different methods have been worn for the synthesis of SnO<sub>2</sub> nanoparticles, such as sol-gel [19], hydrothermal [20], co-precipitation [21], Microwave-assisted methods [22], spray pyrolysis [23]. But the green synthesis of nanoparticles has more attention compared with the aforementioned methods. The main reason behind the green approach for the preparation of nano metal oxides is getting hold of additional importance such as cost-effective, high yield nanoparticle production, etc., compared with other methods [24, 25]. There are limited papers reported studies of green synthesis of SnO<sub>2</sub> using plant extracts as capping and reducing agents towards photocatalytic applications.

The *Delonix elata (D. elata)* tree belongs to *Caesalpinoideae* family. The flowering timbers are the genus members. It has been explored for many pharmaceutical applications as the leaves and bark of this plant is known to have medicinal properties [26]. It is found all over south India. *D. elata* leaf extract contains unique phenolic compounds mainly two flavanones namely Quercetin and Rutin [27]. Table 1 summarizes the compound details which were identified from *D. elata* leaf extract.

In this present study, the authors report the most economical and easiest green synthesizes protocol to prepare  $SnO_2$  nanoparticles using *D. elata* leaf extract as a chelating agent. The three different methods i.e., wet chemical, sonication, and microwave methods were influenced to prepare  $SnO_2$  nanoparticles. The motive for taking these three different processing methods is due to its simplicity and also simply a scalable attribute. Along with it, all three processes reveal an excellent ability of conserve the chemical precursor i.e., bulk production of nanoparticles can be achieved from the very small amount of the starting precursor. Even though the processes seemed quite similar yet they are extremely different in terms of its processing parameters and play a dominant role in particle size, purity, and crystalline nature, morphology, specific surface area of nanomaterials [28]. The influences of these preparation methods on structural, morphological, optical, and photocatalytic properties of SnO<sub>2</sub> nanoparticles were comprehensively discussed. The prepared SnO<sub>2</sub> nanoparticles were proceeding for Rhodamine B (RhB dye) degradation under UV light irradiation.

# 2 Materials and methods

# 2.1 Preparation of D. elata leaf extract

The *D. elata* leaves were collected from villages in Namakkal district, Tamil Nadu, India. The collected leaves were washed with deionized water (DI) to remove the moisture present on it. After washing, the leaves were allowed to dry at room temperature for two days. The dried leaves were powdered by using a domestic mixer (mixie) and then the 10 g of the powdered leaf was dispersed in 300 mL of DI water and soaked in Erlenmeyer flask. The dispersion was heated at 70 °C for 30 min. The leaf extract is been allowed to cool to room temperature without disturbances, and filtered with the help of Whatmann No. 1 filter paper. The final filtrate was stored for further experimental use.

Table 1 Chemical compounds from the extract of *D. elata* leaf (Adapted from Ref. [27])

S. No	Name of the compounds	Structure of the compound	Molecular formula C <sub>15</sub> H <sub>10</sub> O <sub>7</sub>	
1	Quercetin	HO CONTRACTOR		
2	Rutin		C <sub>27</sub> H <sub>30</sub> O <sub>16</sub>	
		HOHO HO		

## 2.2 Synthesis of SnO<sub>2</sub> nanoparticles

The  $\text{SnO}_2$  nanoparticles were green synthesized using the leaf extract of *D. elata* through three different synthesis routes namely i.e., Sonication, wet-chemical, and microwave methods. The base solution was prepared by adding 2.256 g of tin chloride (0.1 M) precursor, (an analytical grade (AR) from Merck, India) to 100 ml of *D. elata* leaf extract and stirred for 24 h at constant rpm to synthesize  $\text{SnO}_2$  nanoparticles. Hereafter this solution is called as a base solution for the preparation of  $\text{SnO}_2$  nanoparticles. All the AR grade chemicals were utilized without any further purification. The synthesis parameters for the aforementioned synthesis routes were employed to a base solution. The synthesis protocol for  $\text{SnO}_2$  nanoparticles using *D. elata* leaf extract is shown in Fig. 1.

#### 2.2.1 Wet-chemical method

For synthesizing  $\text{SnO}_2$  nanoparticles through the wetchemical route, the base solution was agitated vigorously using a magnetic pellet under elevated temperature (70 °C) for 1 h, and then the precipitate was formed. Then, the base solution was dried at 100 °C for 12 h, followed by centrifuge at 5000 rpm, washed several times with DD water, and ethanol, finally, obtained products was calcined at 400 °C for 2 h. The calcined powder i.e.,  $\text{SnO}_2$ nanoparticles was used for further characterization. Now the procured  $\text{SnO}_2$  nanoparticles were termed as  $\text{SnO}_2$ -WC nanoparticles.

#### 2.2.2 Sonication method

The sonication process at 40 kHz (Advanced Sonicator, Lark, New Delhi, India) was employed over the prepared base solution for 1 h, till a clear suspension was obtained. Following the suspension of the precipitates in the base

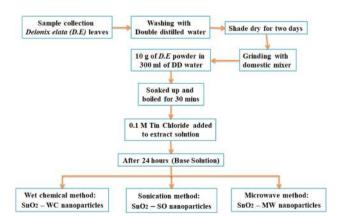


Fig. 1 Synthesis protocol for SnO<sub>2</sub> nanoparticles

solution, it was dried in a hot air oven at 100 °C for 12 h. The dried precipitate was collected and centrifuged at 5000 rpm, washed several times with double distilled water and absolute ethanol, then calcined at 400 °C for 2 h, to produce SnO<sub>2</sub> nanoparticles which were exploited for further characterization studies [29]. The obtained SnO<sub>2</sub> nanoparticles are termed as SnO<sub>2</sub>–SO nanoparticles.

#### 2.2.3 Microwave method

In a typical synthesis of the microwave method, the base solution was taken in Teflon lined vessel which was subject to microwave radiation power (320 W) at constant temperature 100 °C for 15 min and then allowed to cool at room temperature. Final yellow-colored products were collected, centrifuged at 5000 rpm, washed multiple times with double distilled water, and absolute ethanol which is kept in an oven at 100 °C for 3 h. The SnO<sub>2</sub> nanoparticles were then obtained by calcinations of as-prepared powder in air at 400 °C for 2 h. The procured SnO<sub>2</sub> nanoparticles are termed as SnO<sub>2</sub>–MW nanoparticles.

## 2.3 Characterisation techniques

The X-ray diffraction patterns of the prepared SnO<sub>2</sub> nanoparticles were obtained by using a powder X-ray diffractometer (XRD; X'Pert PRO, PANalytical, Almelo, the Netherlands) operated with the Cu at 40 kV and 30 mA. The XRD pattern was analyzed in the  $2\theta$  range from 20 to  $80^{\circ}$ . The Fourier transforms infrared (FTIR) spectra of the SnO<sub>2</sub> nanoparticles were recorded using an FTIR spectrophotometer (Spectrum 100; PerkinElmer, USA) in the range of 4000-450 cm<sup>-1</sup> using KBr as an active medium by mixing 200:1 ratio of KBr. The particle size analyzer (Nanophox Sympatec Germany) used to decide the common particle size distribution primarily based on the dynamic light scattering (DLS) method and laser light of wavelength 633 nm. It was measured in the range from 1 to 1000 nm at a scattering angle of 90°. The measurement was continuously repeated for five-times to get average particle size. The average particle size of SnO<sub>2</sub> nanoparticles was calculated by using the automated mode. The UV-visible (UV-vis) spectrophotometer (Agilent Cary 8454, Singapore) was used to determine the absorbance capability of the prepared SnO<sub>2</sub> nanoparticles with distilled water as a solvent for a wide range from 180-800 nm wavelength of electromagnetic spectral region. The specific surface area (SBET) was calculated using the Brunauer-Emmett-Teller (BET) process using a BET surface area analyzer (Autosorb AS-1MP; Quantachrome, Boynton Beach, FL). The sample was degassed under vacuum at 363 K for 3 h to remove the physisorbed moisture. The physisorption study was carried out with N<sub>2</sub> adsorption-desorption measurements

under liquid N<sub>2</sub> temperature. The mean pore size distributions and total pore volume were calculated using the Barrett–Joyner–Halenda (BJH) method. The thermal degradation properties of the prepared SnO<sub>2</sub> nanoparticles were evaluated by using Thermogravimetric analyzer and Differential thermal analyzer (Exstar TG/DTA 6300, Hitachi, Tokyo, Japan) with a heating rate of 10 °C/min up to 1000 °C at nitrogen atmosphere.

#### 2.4 Photocatalytic degradation activity

The photocatalytic dye degradation activity of the green synthesized SnO<sub>2</sub> nanoparticles was carried out by assessing the degradation of the RhB dye in an aqueous solution under constant UV light irradiation. In the photocatalytic degradation process, a standard volume of RhB dye was taken and 20 mg of each SnO<sub>2</sub> nanoparticles were added to RhB dye solution [30]. The photoreaction was performed in a cubic UV reaction chamber having an annular UV illumination. Irradiation was provided using a 40 W UV lamp with major emission at 365 nm, located at the corners of the UV reactor (Agilent, Singapore). With the effect from UV light irradiation, for every 30 min once, 5 ml of the aqueous solution was collected from the UV reaction chamber and subjected to measure the UV absorbance range of RhB dye at  $\lambda_{max}$  = 556 nm. The photocatalytic dye degradation efficiency of the prepared SnO<sub>2</sub> nanoparticles was calculated by using the following Eq. (1) [29]

$$\eta = \left[\frac{C_0 - C_t}{C_0}\right] \times 100\% \tag{1}$$

Here,  $\eta$  is the degradation percentage,  $C_0$  is the initial absorbance of dye (at 00 min);  $C_t$  is the change in absorbance of the dye at after time intervals of the degradation.

### **3** Results and discussion

#### 3.1 Fourier transform infrared spectra (FTIR)

#### 3.1.1 FTIR spectra of D. elata leaf extract

The FT-IR study of *D. elata* leaf extract is shown in Fig. 2. The functional groups of the organic and functional compounds of the leaf extract were determined. Infrared spectrum shows band area 3420 cm<sup>-1</sup> is the presence of O–H groups of leaf extract. The peak area at 2920 cm<sup>-1</sup> is related to vibration C–H ring stretching. The band area at 2860 cm<sup>-1</sup> shows symmetric C–H stretching. A strong stretching vibration at 1630 cm<sup>-1</sup> shows the presence of carbonyl (C=O) groups. The absorption band at

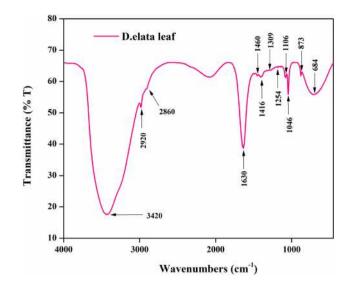


Fig. 2 FTIR spectra of D. elata leaf extract

1460 cm<sup>-1</sup> corresponds to vibrations of NH plane bending. The band area a 1416 cm<sup>-1</sup> shows CH<sub>2</sub> bending.

The peak at 1309 cm<sup>-1</sup> indicated by the presence of alkyl ketone components. The band area 1254 cm<sup>-1</sup> shows the sulphonic acid esters (SO<sub>3</sub>). 1106 cm<sup>-1</sup> band area is showing C–O–C aryl conjugated group. The band at 1046 cm<sup>-1</sup> shows C–N stretching alkyl amine and peak at 873 cm<sup>-1</sup> show the C–H out plane bending. The band areas from 684 to 450 cm<sup>-1</sup> shows halogen compounds like C–Cl, C–Br, and C–I compound [31].

The polyhydroxyl compounds namely Quercetin and Rutin exiting in the *D. elata* leaf extract act as ligation agents and the aromatic hydroxyl groups of Rutin react readily with tin ions, which lead to the formation of a stable complex of  $SnO_2$  nanoparticles [27]. The complex was decomposed at the calcination process which gave rise to  $SnO_2$  NPs. The possible phytochemical reaction during the synthesis process of  $SnO_2$  nanoparticles using *D. elata* leaf extract is given in Fig. 3 [32].

#### 3.1.2 FTIR spectra of SnO<sub>2</sub> nanoparticles

The FT-IR spectra were shown in Fig. 4a–c respectively for  $SnO_2$ –WC,  $SnO_2$ –SO,  $SnO_2$ –MW nanoparticles. The FTIR spectra of  $SnO_2$  nanoparticles have the major bands at 3434 cm<sup>-1</sup>, 2920 cm<sup>-1</sup>, 2848 cm<sup>-1</sup>, 1618 cm<sup>-1</sup>, 1381 cm<sup>-1</sup>, 608 and 506 cm<sup>-1</sup> [33]. The broad band around at 3365 to 3620 cm<sup>-1</sup> and 1618 cm<sup>-1</sup> due to stretching vibrations of Sn–OH groups and due to O–H stretching vibrations of water molecules absorbed from the environment by  $SnO_2$  nanoparticles and which may present in the surface of the  $SnO_2$  nanoparticles [33]. The band area around 2848 and 2920 cm<sup>-1</sup> indicates the presence of C–H group. The



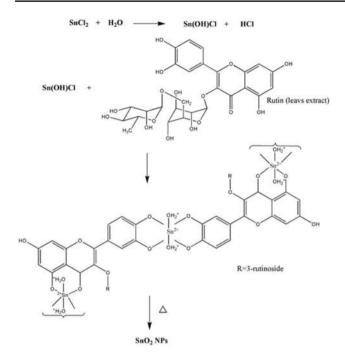


Fig. 3 Formation of SnO<sub>2</sub> nanoparticles

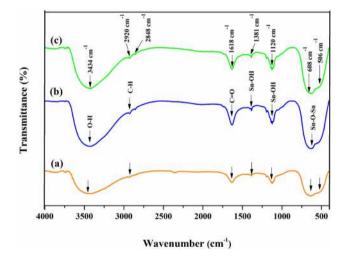


Fig. 4 shows FTIR Spectra of a SnO2–WC, b SnO2–SO, and c SnO2– MW nanoparticles

peak area around 500 to 650 cm<sup>-1</sup> is ascribed to the Sn–O stretching vibration and the O–Sn–O bending vibration, respectively. For the above reason, the existence of these bands confirmed the formation of SnO<sub>2</sub> nanoparticles [33, 34].

#### 3.2 X-ray diffraction patterns (XRD)

The XRD patterns of  $SnO_2$  nanoparticles were represented in Fig. 5. The XRD studies confirmed the presence

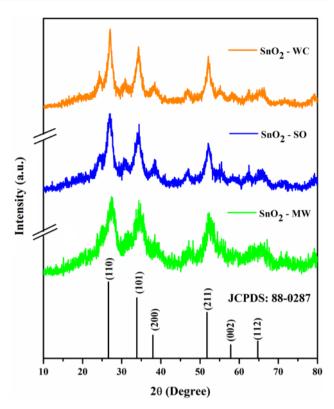


Fig. 5 XRD pattern for SnO<sub>2</sub> nanoparticles

of high crystalline SnO<sub>2</sub> nanoparticles which was perfectly matched with standard JCPDS No.: 88-0287 for all the SnO<sub>2</sub> nanoparticles but with slight distortion in diffraction angle (2 $\theta$ ) and intensities of the XRD peaks. The diffraction angle values obtained from XRD pattern for SnO<sub>2</sub> nanoparticles are 26.90°, 34.21°, 37.04°, 52.16°, 58.36°, and 65.84°, which are indexed with the (110), (101), (200), (211), (002), (112) planes, respectively. However, it was necessary to note that the intensity of the diffraction peaks shows an increasing trend with a change in synthesis methods. The crystalline SnO<sub>2</sub> nanoparticle formation is high for microwave influenced method compared with the other two methods. The following Debye Scherer Eq. (2) used to determine the crystallite size [35, 36],

$$D = \frac{k\lambda}{\beta \text{Cos}\theta} \tag{2}$$

where k is constant (0.9),  $\lambda$  is the wavelength having value 1.540 Å corresponding to Cu K $\alpha$  source,  $\theta$  is the Bragg angle and  $\beta$  is the full width of the half maximum.

The calculated crystallite size of  $SnO_2$  nanoparticles was found to be 7.61, 6.09, and 5.92 nm respectively for  $SnO_2$ -WC,  $SnO_2$ -SO, and  $SnO_2$ -MW nanoparticles. The various structural parameters like lattice parameters, unit cell volume, dislocation density and microstrain values of SnO<sub>2</sub> nanoparticles were calculated by using the following relations and estimated values were tabulated in Table 2.

$$\frac{1}{d^2} = \frac{h^2 + k^2}{a^2} + \frac{l^2}{c^2}$$
(3)

$$V = a^2 \cdot c \tag{4}$$

$$\delta = \frac{1}{D^2} \tag{5}$$

$$\varepsilon = \frac{\beta}{4\tan\theta} \tag{6}$$

#### **3.3** Thermal analysis of SnO<sub>2</sub> nanoparticles

TGA profile curves of the prepared SnO<sub>2</sub> nanoparticles are shown in Fig. 6. Three major weight losses can be observed in the TGA curve at 90 °C (I), 380 °C (II), and 470 °C (III). The minor weight loss (I) in the low-temperature region indicates the presence of a water molecule of the lattice. It is observed that the TGA curve for SnO<sub>2</sub>–WC and SnO<sub>2</sub>–SO exhibit a weight loss of 25.1 and 24.06% occurred in the temperature range from 300 to 700 K but for SnO<sub>2</sub>-MW exhibits 20.51% of weight loss only observed in TGA curve. These results clearly indicate the microwave irradiation makes high thermally stable SnO<sub>2</sub> nanoparticles when compared with the other two methods. Thus the weight losses are due to the water evaporation and decomposition of organic components present in the prepared  $SnO_2$ nanoparticles which may come from D. elata leaf extract. Similar weight loss observation of green synthesized SnO<sub>2</sub> nanoparticles are already reported [8].

#### **3.4** Morphological studies of SnO<sub>2</sub> nanoparticles

Figure 7a, c, and e shows the FESEM images of  $SnO_2$ -WC,  $SnO_2$ -SO, and  $SnO_2$ -MW nanoparticles. In Fig. 7a, c, and e shows clusters like foam morphology with small agglomerations for all  $SnO_2$  nanoparticles but  $SnO_2$ -MW (Fig. 7e) exhibits the uniform distribution morphology it may be formed due to effects of microwave irradiation during

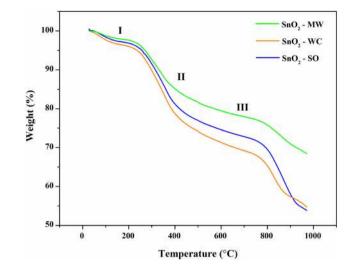


Fig. 6 shows the Thermogravimetric analysis of green synthesized  ${\rm SnO}_2$  nanoparticles

the synthesis process. The high surface to volume ratio of  $SnO_2$ -MW nanoparticles may be due to their uniform distributions and very small-sized cluster like foam morphology in its crystalline and UV absorptions natures [37, 38]. Figure 7b, d, and f shows the EDX spectra of prepared  $SnO_2$  nanoparticles. The EDX spectrum validates that all prepared  $SnO_2$  nanoparticles consist only Sn and O with a very small amount of Cl. The presence of Cl peak in the EDX spectrum may be due to the residue of starting precursor which can be easily removed by undergone through a water wash. Thus, the purity of the prepared  $SnO_2$  nanoparticles is very high. The EDX quantitative results of  $SnO_2$ nanoparticles are given as an inset table in EDX images (Fig. 7b, d, and f).

#### 3.5 Particle size analysis

Figure 8 shows the average particle size distribution of the prepared  $SnO_2$  nanoparticles. The average particle size (d<sub>50</sub>) of the prepared  $SnO_2$  nanoparticles are found around 13–18 nm. It is in the range of 9.4 (d<sub>10</sub>)–37.41 (d<sub>90</sub>), 9.0 (d<sub>10</sub>)–33.41 (d<sub>90</sub>), and 6.8 (d<sub>10</sub>)–27.2 (d<sub>90</sub>) nm size, respectively, for  $SnO_2$ –WC,  $SnO_2$ –SO, and  $SnO_2$ –MW

Sample name	Crystallite size (nm)	Lattice param- eter (Å)		Unit cell volume (ų)	Dislocation density 'δ'	Micro strain ' $\epsilon$ ' (×10 <sup>-3</sup> /lines <sup>2</sup> m <sup>4</sup> )	
		a=b	с		(×10 <sup>15</sup> lines/ m <sup>2)</sup>		
SnO <sub>2</sub> –WC	7.61	4.659	3.129	67.95	0.1727	6.6871	
SnO <sub>2</sub> –SO	6.09	4.717	3.140	69.89	0.2696	4.6579	
SnO <sub>2</sub> –MW	5.92	4.677	3.202	70.07	0.2853	3.0324	
JCPDS No: 88-0287	4.737	3.186	71.51	-	-		

Table 2Structural parametersof SnO2 nanoparticles obtainedfrom XRD analysis

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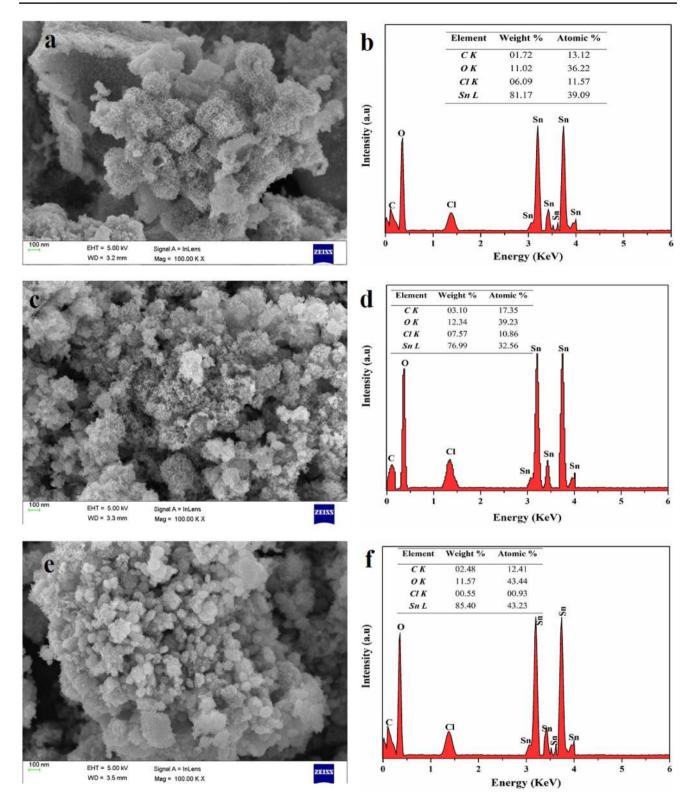


Fig. 7 shows FESEM images (a, c, & d) and EDX pattern of (b, d, & f) SnO<sub>2</sub>–WC, SnO<sub>2</sub>–SO, and SnO<sub>2</sub>–MW nanoparticles

nanoparticles. Moreover, the mean particle size distribution ( $d_{50}$ ) of SnO<sub>2</sub> nanoparticles is 18.75 ± 3, 17.40 ± 3, and 13.68 ± 3 nm, respectively, for samples SnO<sub>2</sub>–WC, SnO<sub>2</sub>–SO,  $SnO_2$ -MW nanoparticles. It is found that prepared all the  $SnO_2$  nanoparticles influence the formation of particles at nanoscale under different processing methods. The

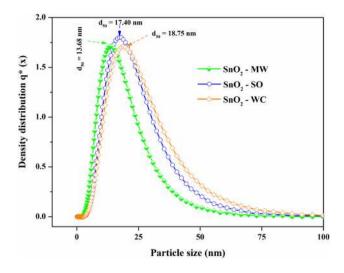


Fig. 8 shows Particle size distribution curve of SnO<sub>2</sub> nanoparticles

 $SnO_2$ -MW exhibit a smaller particle size compared to  $SnO_2$ -WC and  $SnO_2$ -SO, which is also evidenced from the obtained crystalline peaks of XRD.

#### 3.6 Specific surface area analysis

The N<sub>2</sub> adsorption–desorption isotherm of prepared  $SnO_2$  is shown in Fig. 9a and b. The observed BET surface area, pore size, and pore volume of  $SnO_2$  nanoparticles are given in Table 3. The  $SnO_2$  nanoparticles displaced type-IV isotherm from desorption according to the IUPC nomenclature, but with a major change in the desorption isotherm for all  $SnO_2$  nanoparticles due to particle

 Table 3
 Nitrogen sorption porosimetry studies and average particle size distributions of SnO<sub>2</sub> nanoparticles

S. No.	Sample	S <sub>BET</sub>	Pore vol- ume (cm <sup>3</sup> g <sup>-1</sup> )	Mean pore size (nm)	Particle size (nm) from DLS
1	SnO <sub>2</sub> –WC	101	0.5813	15.24	18.75
2	SnO <sub>2</sub> –SO	169	0.7709	05.29	17.40
3	SnO <sub>2</sub> –MW	196	1.0081	02.96	13.68

size. In the case of  $SnO_2$ –MW and  $SnO_2$ –SO, the amount of N<sub>2</sub> adsorption rapidly increases up to a relative pressure of 0.4 beyond which a hysteresis loop is adsorbed in the relative pressure ranges from 0.1 to 0.9. The above observation clearly demonstrated that the existence of mesopores nature as shown in Fig. 9b.

The BET surface area of SnO<sub>2</sub>-MW nanoparticles is very high (196  $m^2g^{-1}$ ) compared with the SnO<sub>2</sub>-SO (169  $m^2q^{-1}$ ), SnO<sub>2</sub>–WC (101  $m^2q^{-1}$ ) nanoparticles. The observed huge BET surface area for the SnO<sub>2</sub>-MW is due to its lower particle size (13.68 nm) than SnO<sub>2</sub>-SO (17.40 nm), SnO<sub>2</sub>-WC (18.75 nm) nanoparticles. It is betrayed from the above observation that the characteristic properties of SnO<sub>2</sub> nanoparticles treated by three different synthesis methods influence the specific surface area i.e., as the average particle size decreases, the specific surface area of the SnO<sub>2</sub> nanoparticle increases. The observed results confirm the relationship between the surface areas to the particle size of the nanomaterials. These investigations denote that the SnO<sub>2</sub> nanoparticles with lower particle size and high surface area may lead to enhance physical and electrical property [39, 40].

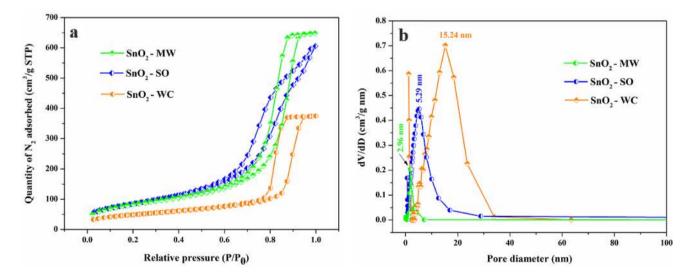


Fig. 9 shows N<sub>2</sub> adsorption – desorption isotherm (a) and BJH pore size distribution curve (b) of SnO<sub>2</sub> nanoparticles

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## 3.7 Optical properties

UV-visible absorption spectra were recorded in order to perceive the optical properties of synthesized  $SnO_2$  nanoparticles. The Fig. 10a represented the UV-vis absorption spectra of  $SnO_2$ -WC,  $SnO_2$ -SO, and  $SnO_2$ -MW nanoparticles. From Fig. 10a it was manifested that broad peaks of infinitesimal intensity commenced beginning around 220 to 260 nm.

It was apparent that the intensity of these broad absorption peaks started to decrease with change in influencing parameters and move towards lower wavelength. The optical properties are strongly depending upon the particle size of the nanoparticles [39, 40]. From the UV-vis spectra, it is evident that the absorption onset shows a blue shift with a decrease in crystallite size. The observed blue shift absorption spectra are the direct consequence of quantum confinement effect which is associated with particle size [35]. Due to the quantum confinement effect, the bandgap of the nanoparticle increases as the particle size decreases and results in the shift of absorption spectra towards a lower wavelength. From the absorption spectra, direct bandgap energy is calculated using Tauc relation which is shown in Fig. 10b. For semiconductor nanoparticles, to relate the absorption coefficient with incident photon energy following equation has been used [4, 35].

$$\alpha(v)hv = K \left(hv - E_a\right)^n$$

 $\alpha(\nu)$  Absorption coefficient, K is a constant, E<sub>g</sub> is energy bandgap,  $h\nu$  is incident photon energy, and n equals to  $\frac{1}{2}$  for direct bandgap semiconductor and equals to 2 for indirect band gap semiconductors.

The calculated energy bandgap values are 3.80, 3.89, and 3.91 eV respectively for  $SnO_2$ -WC,  $SnO_2$ -SO,  $SnO_2$ -MW nanoparticles.

#### 3.8 Photocatalytic degradation of Rhodamine B dye

The highly possible photocatalytic degradation mechanism of the RhB dye by using prepared  $SnO_2$  nanoparticles can be enlightened by the following standard protocol. When the photocatalyst surface is illuminated with photon energy equals or greater than its natural bandgap energy, it will lead to the formation of holes (h<sup>+</sup>) in the valence band and an electron (e<sup>-</sup>) in the conduction band of the prepared  $SnO_2$ nanoparticles. The hole (h<sup>+</sup>) acts as an oxidizing agent and oxidizes the pollutant directly or water to form hydroxyl radicals of degraded products.

$$SnO_{2} + hv \rightarrow e^{-} + h^{+}$$

$$h^{+} + H_{2}O \rightarrow OH + h^{+}$$

$$OH^{-} + h^{+} \rightarrow OH$$

$$O_{2} + e^{-} \rightarrow O_{2}^{-}$$

$$O_{2}^{-} + H^{+} \rightarrow OOH$$

$$RhB + hv \rightarrow RhB^{*}$$

$$RhB^* + SnO_2 \rightarrow RhB^* + SnO_2(e^-)$$

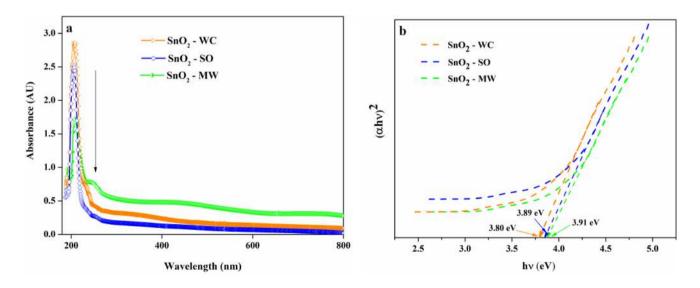


Fig. 10 shows UV-vis absorption spectra and Tauc plot of prepared SnO<sub>2</sub> nanoparticles

 $SnO_2(e^-) + O_2 \rightarrow SnO_2 + O_2^ SnO_2(e^-) + O_2^- + 2H \rightarrow SnO_2 + H_2O_2$ 

 $SnO_2(e^-) + H_2O_2 \rightarrow SnO_{2+}^{,}OH + OH^-$ 

 $RhB^* + O_2 \text{ or } O_2^- \text{ or } OH \rightarrow degradation compounds$ 

Figure 11a–c shows the photocatalytic degradation process of RhB dye under UV light irradiation for constant time intervals respectively for without  $SnO_2$ ,  $SnO_2$ –WC,  $SnO_2$ –SO,  $SnO_2$ –MW nanoparticles. After 30 min of UV irradiation, the absorbance peaks of RhB dye strongly decreased when compared with pure  $SnO_2$ and RhB dye with the addition of  $SnO_2$  nanoparticles.

The results announcing the degradation of RhB dye with the addition of  $SnO_2$  nanoparticles under UV light radiation. The photocatalytic degradation efficiency of  $SnO_2$  nanoparticles was calculated by using Eq. 1. The degradation efficiency of the  $SnO_2$ –WC,  $SnO_2$ –SO, and  $SnO_2$ –MW nanoparticles after 150 min of UV light irradiation are 82.3%, 85.6%, and 92.8% respectively and 5.6% for RhB dye without  $SnO_2$  nanoparticles. The degradation results were clearly indicated that the prepared  $SnO_2$ –MW nanoparticle exhibits the improved degradation efficiency compared with  $SnO_2$ –WC, and  $SnO_2$ –SO nanoparticles.

Its crystalline nature with high in purity, uniformly distributed clusters like foam morphology with higher surface area helps to exhibit better degradation results while

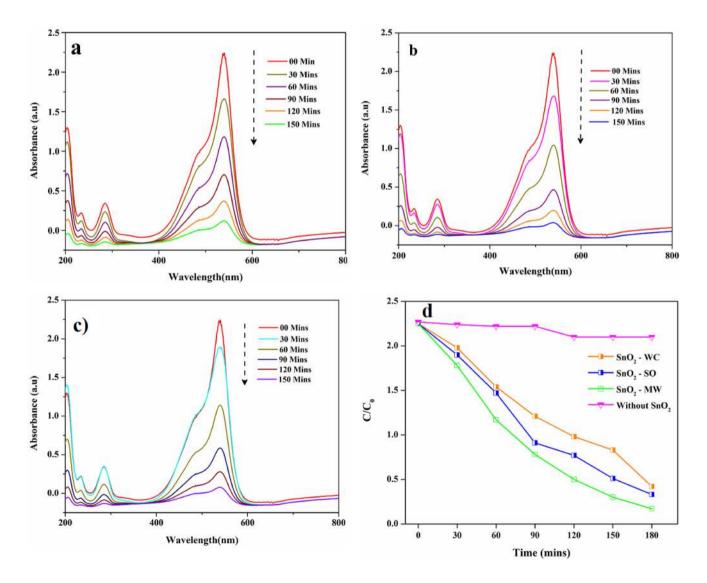


Fig. 11 depicts Photocatalytic responses of a SnO<sub>2</sub>–WC, b SnO<sub>2</sub>–SO, c SnO<sub>2</sub>–MW nanoparticles and d degradation efficiency under UV light irradiation

SN Applied Sciences

compared with earlier reports [42]. The obtained photocatalytic degradation performances of the prepared SnO<sub>2</sub> nanoparticles were compared with previously published articles which are summarized in Table 4.

After the completion of the degradation process, to confirm the presence of degraded products from RhB dye which were absorbed on the surface of SnO<sub>2</sub> nanoparticles, the photocatalyst was separated from the dye mixture and dried in a hot air oven at 80° C without washing it, and analyzed by FT-IR spectra. The FTIR spectra of SnO<sub>2</sub> nanoparticles after the photocatalytic reaction is shown in Fig. 12. After the degradation process, the intensity of some peaks is reduced and also a few new peaks have been raised while comparing with SnO<sub>2</sub> nanoparticles before the photocatalytic reaction process. These changes are due to the adsorption of degraded compounds from the RhB dye settled on the surface of SnO<sub>2</sub> nanoparticles [41].

# 4 Conclusion

The highly crystalline  $\text{SnO}_2$  nanoparticles were synthesized by novel and eco-friendly routes by employing *D. elata* leaf extract as a reducing agent by influencing three different processes namely wet chemical, sonication, and microwave irradiation. The microwave irradiation influenced  $\text{SnO}_2$  nanoparticles shows ( $\text{SnO}_2$ -MW) excellent crystallinity and nanoparticles exhibit higher surface area 196 m<sup>2</sup>g<sup>-1</sup> compared with the other two methods. Due to the influencing process, particle sizes of the prepared  $\text{SnO}_2$  nanoparticles are gradually decreased. The microwave irradiated  $\text{SnO}_2$  nanoparticles show the excellent photocatalytic degradation

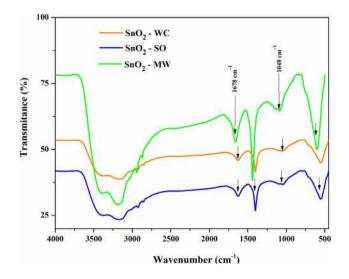


Fig. 12 shows FTIR Spectra of  ${\rm SnO}_2$  nanoparticles obtained after the degradation process

behaviour compared with the other two method influenced  $SnO_2$  nanoparticles. It shows 90.8% of degradation efficiency in lesser than 150 min of UV light irradiation while 80%, 83.2% of degradation efficiency given by  $SnO_2$ -WC and  $SnO_2$ -SO nanoparticles. From the above brief experimental assessments, a green synthesized  $SnO_2$  nanoparticle by microwave-assisted method is a potential candidate for pollutant dye degradation applications. In addition, this comparative assessment also helps to understand the influence of the processing method on the green synthesized  $SnO_2$  nanoparticles which in turn helps in the tuning of nanoparticles as per the multifunctional applications.

Table 4	the comparative assessment o	f photocatalyt	ic activity of	prepared SnO <sub>2</sub>	nanoparticles with	existing reports

Name of the source	Part of the source	Photocatalyst	Dye	Dye con- centration (g)	Reaction time (mins)	Degradation efficiency (%)	References
Delonix elata	Leaf	SnO <sub>2</sub> –MW	Rhodamine B	0.2	150	92.47	This work
		SnO <sub>2</sub> –SO	Rhodamine B	0.2	150	85.61	
		SnO <sub>2</sub> –WC	Rhodamine B	0.2	180	81.30	
C.betacea	Fruit	$SnO_2$	Methylene blue	0.5	70	85.1	[42]
Calotropis gigantea	Leaf	SnO <sub>2</sub>	Methyl orange	0.2	180	80.4	[43]
Psidium Guajava	Leaf	SnO <sub>2</sub>	RY186 dye	0.3	180	88.9	[44]
cauliflower	Leaf	$SnO_2$	Methylene blue	0.2	180	88.2	[45]
sugar cane	sugar cane	SnO <sub>2</sub>	Rose Bengal	0.2		87.9	[46]
			Methylene blue	0.2	180	85	
Bacteria	Erwinia herbicola	$SnO_2$	Methylene blue	0.2	160	93	[47]
			Methylene orange	0.2	160	93	

# **Compliance with ethical standards**

**Conflict of interest** The authors declare that they have no conflict of interest.

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