



CHARACTERIZATION OF ZNO NANOPARTICLES PREPARED IN *CURCUMA AROMATICA SALISB.* ROOT EXTRACT

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ABSTRACT

The root extract of the locally available *Curcuma Aromatica Salisb.* was used with the ZnO precursors to study the effect of the *Curcuma Aromatica Salisb.* extract on the growth and optical absorption behaviour of the ZnO nanoparticles. ZnO nanoparticles were synthesized in various concentrations of *Curcuma Aromatica Salisb.* root extract. X-ray diffraction studies show that the prepared samples consist of nanocrystallites of having sizes in the range between 17 nm to 26 nm. The optical absorption studies reveal that the eco-dyeing on ZnO nanoparticles using *Curcuma Aromatica Salisb.* root extract has enhanced the optical absorption behaviour of ZnO to the visible region of the spectrum.

Keywords: ZnO nanoparticles; *curcuma aromatica salisb*; eco-dyeing.

INTRODUCTION

Curcuma aromatica Salisb., the wild turmeric, 'Vanaharidra' in Ayurveda, belongs to the 'ginger family' Zingiberaceae. This wild turmeric is an aromatic medicinal plant and is commonly known as "kasturi manjal" (musk turmeric) in south India. The rhizome of the plant is loaded with alkaloids, flavonoids, curcuminoids, tannins and terpenoids. The medicinal properties of this rhizome are being used in many traditional systems of medicines. The rhizomes have characteristic fragrance. The paste of rhizome is used for facial application to reduce acne and excessive hair growth and also to improve skin tone and complexion by village women in South India^[1-3]. The colouring matter in the rhizomes of *Curcuma aromatica Salisb.* is predominantly curcumin and demethoxycurcumin^[4]. Revathi et al., studied the antibacterial activity of the rhizome of *Curcuma aromatica Salisb.* and reported that the hexane extract of the rhizome of *Curcuma aromatica Salisb*^[5]. contains the compounds such as Aromadendrene, a-Vatrenene, Epiglobulol, Germacron,

Xanthinin, 2-Nonadecanone, Octadecanoic acid, 1-2-benzenedicarboxylic acid-diisooctyl ester etc.,^[5].

Eco-dyeing or natural colouring of the nanoparticles for enhancing their absorption characteristics is an important field of research for developing electrode materials for Dye Sensitized Solar Cells (DSSCs). In DSSCs, the incident light causes the photoexcitation of the dye molecules which subsequently injects electrons into the conduction band of the semiconducting material. In this work, we have chosen ZnO for the eco-dyeing and the *Curcuma aromatica Salisb.* as an eco dye or natural colouring materials. ZnO is chosen because of its wide bandgap (3.37 eV), large exciton binding energy (60 meV), high electron mobility, piezoelectric and pyroelectric properties, biocompatibility, non-toxic nature, very high specific surface area, good chemical and thermal stability^[6]. Zinc oxide (ZnO) nanoparticles are promising material due to their unique and multifunctional properties and is used to create photoanodes for Dye-Sensitized Solar Cells (DSSCs).

The main objective of this paper is to discuss the potential use of the *Curcuma aromatica Salisb.* to sensitize the ZnO nanoparticles and to study their characteristics. There are lots of reports available on green synthesis of ZnO nanoparticles using plant leaf, root, stem, flower and seed extracts, but no reports available on the synthesis of nanoparticles in *Curcuma aromatica Salisb.* and of eco-dyeing of nanoparticles. Green synthesis of ZnO nanoparticles and eco-dyeing are different in the process itself. In the case of green synthesis, especially based on plant extracts, the extracts are utilized to reduce and stabilize the nanoparticles. The phytochemicals present in plant extracts have the potential to reduce metal ions in a much shorter time when compared to other reducing agents. The main phytochemicals present in plants are flavanoids, terpenoids, sugars, ketones, aldehydes, carboxylic acids, and amides, which are responsible for bioreduction of nanoparticles^[4]. But in the eco-dyeing process, natural dyes are used with the purpose of surface modification to change their optical behavior not to control their particle size. The optical absorption and surface characteristics of eco-dyed ZnO nanoparticles are discussed here.

METHODS

Curcuma aromatica Salisb. powder were obtained from local ayurvedic shop and the extracts were obtained in deionized water. Different concentrations of the *Curcuma aromatica Salisb.* extracts were prepared in 10 ml of deionized water. Then the extract was filtered to remove the residues and impurities and the filtrate is used for further experiments. Zinc acetate dihydrate ($\text{Zn}(\text{CH}_3\text{COO})_2 \cdot 2\text{H}_2\text{O}$) and sodium hydroxide (NaOH) of 99.95% purity were purchased from Sigma-Aldrich Chemicals and used without further purification.

20 ml of 0.75 M zinc acetate dihydrate was prepared in de-ionized water then 10ml of the prepared *Curcuma aromatica Salisb.* solution of different concentrations were added slowly to the 0.75 M zinc acetate dihydrate solution under magnetic stirring. The mixture was stirred for 10 minutes by keeping the hotplate at 70°C. while stirring, 20 ml of 1M NaOH solution added dropwise to the mixture of zinc acetate dihydrate and tannin using a burette. A precipitate of ZnO was obtained and the sodium acetate formed during the reaction process is soluble in water, that can be removed by repeated washing. The supernatant is removed using a syringe and the precipitate was washed many times by continuing the syringe method. After, repeated washing, the obtained precipitate was dried and the resultant powder was collected for analysis.

X-ray diffraction (XRD) analysis of the powder samples were done at wavelength 0.1546 nm, running at 40 kV and 30 mA in X-ray diffractometer (Bruker D8 Advance). X-ray

diffractograms of zinc oxide nanoparticles were recorded in the region from 10° to 80° at a scan speed of 2° per minute. For UV spectroscopic analysis, prepared powder of ZnO nanoparticles were dispersed in distilled water (1 mg/ mL) and scanned in Agilent Cary 5000 UV-vis spectrometer at 25°C in the range of 250–900nm. Fourier Transform Infrared (FTIR) analysis of the ZnO were done by mixing ZnO nanoparticles and potassium bromide to form a salt plate. Spectra between 4000 and 400 cm^{-1} were recorded using a Thermo Nicolet Avatar 370 spectrometer.

RESULTS AND DISCUSSION

Curcuma aromatica Salisb. solution of various concentrations (0.2, 0.4, 0.6, 0.8, 1.0 and 1.2 mg per 10 ml of deionized water) were used to enhance the absorption characteristics of the ZnO nanoparticles. *Curcuma aromatica Salisb.* solution consists of many number of organic compounds^[5]. The extracts confirmed the presence of phenolic (–OH) groups^[7] and the FTIR spectrum of the solution is shown in Figure 1. The analysis was performed in a frequency range of 4000 – 400 cm^{-1} at room temperature. The observed peaks and corresponding vibrations are tabulated in the Table 1. The O-H stretch band around 3400 cm^{-1} is associated with the moisture. From the intensity of the absorption, it is inferred that the 0.8T sample has little higher moisture than other samples. The spectrum presented a band at around 890 cm^{-1} and around 424 cm^{-1} signals the characteristic bond of Zn – O, which confirms the presence of zinc oxide in the sample.

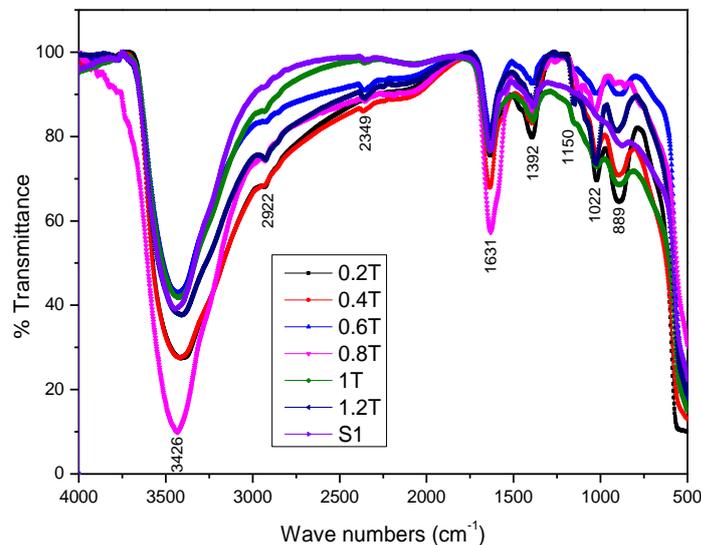


Figure 1. FTIR Spectra of the as synthesized and *Curcuma aromatica Salisb.* dyed ZnO nanoparticles.

Table 1. FTIR bands and corresponding vibrations of eco-dyed ZnO using *Curcuma aromatica Salisb.*

S1	0.2T	0.4T	0.6T	0.8T	1T	1.2T	Vibrations
3439	3413	3418	3433	3433	3421	3416	O-H stretching vibration
-	2922	2924	2926	2928	2923	2917	C-H stretching vibration
-	2349	2345	2342	2350	2343	2341	O-H stretching vibration
1633	1634	1636	1633	1632	1636	1336	C=C stretching vibration
1386	1393	1393	1391	1389	1384	1388	CH ₃ sym. bending
-	1150	1149	-	1120	-	1151	C-O-C stretching vibration
-	1021	1022	1023	1033	1022	1026	C-O stretching vibration
869	892	896	890	893	898	907	ZnO
424	478	403	433	433	437	428	ZnO stretching ^[8]

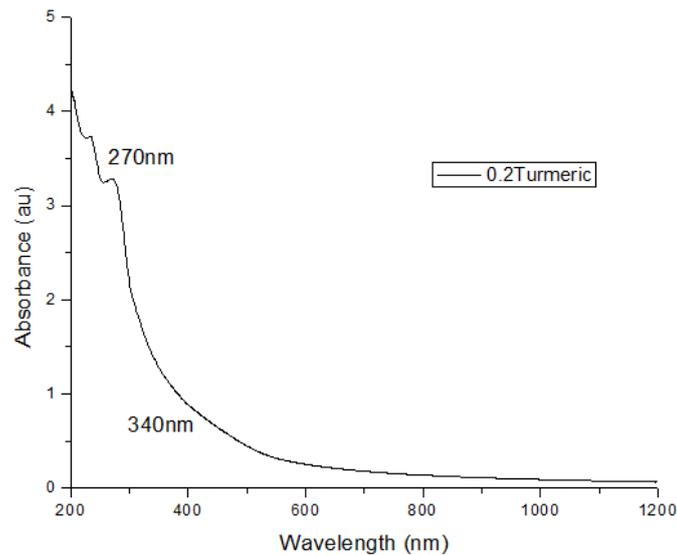


Figure 2. UV-visible absorption spectrogram of the *Curcuma aromatica Salis b.* liquid sample

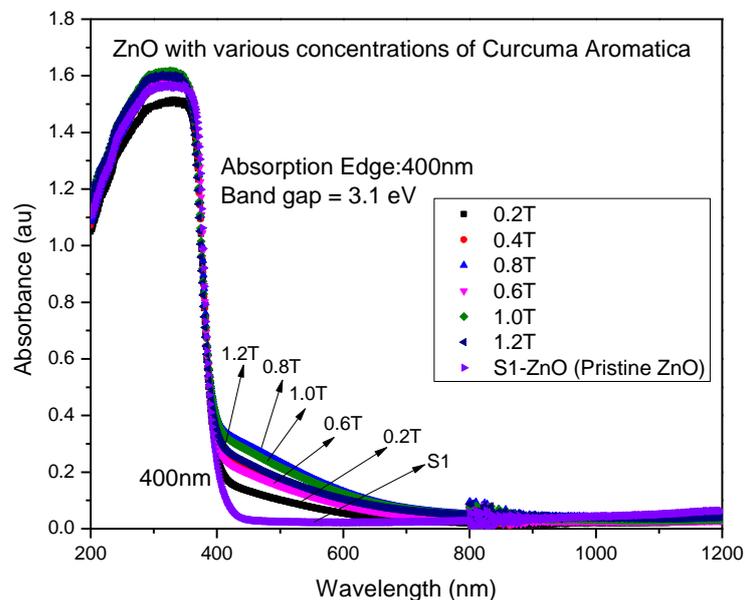


Figure 3. UV- visible absorption spectra of the pristine ZnO and the ZnO nanoparticles prepared in *Curcuma aromatica Salis b.*

The UV-visible absorption spectra of the prepared liquid sample of the concentration 0.2T was obtained as given in the Figure 2. It is observed that the *Curcuma aromatica Salis b.* has absorption peak around 270 nm and absorption edge around 340 nm. Neerja et al. reported UV absorption peaks at around 240,420 nm for curcumin 251 and 423 for demethoxycurcumin^[4]. The observed spectrum of our *Curcuma aromatica Salis b.* solution shows that the samples have absorption in the visible region and the solution were seen as yellowish in color. This absorption in the visible region helps us to sensitize ZnO nanoparticles to expand ZnO spectrum to visible region. Also, the broadening of the

absorption spectra is desirable for harvesting the solar spectra in visible region that leads to a higher photocurrent from the dye sensitized solar cell. Then the absorption spectra of the prepared ZnO nanoparticles powder were studied and the spectra is shown in the Figure 3. The absorption edge observed at 400 nm (3.1 eV) for the pristine ZnO white powder. But, the absorption spectra of the ZnO prepared in the various concentrations of the tamarind tannin extended to visible region (650 nm) with the same absorption peak seen in the spectrum of the *Curcuma aromatica Salisb.* solution alone. The absorption edge due to the ZnO in the eco-dyed sample has not moved remarkably from the position of the pristine sample, it means that the size of the ZnO has not much changed when they are prepared in the presence of *Curcuma aromatica Salisb.*

The band gap of the prepared powder material has been determined by the modified tauc plot method proposed by Ragib Ahsan et al [9].

For preparing Tauc plot for nanoscale powders, Ragib Ahsan et al [9] assumed that

$$\frac{\Delta I}{I_0} = \alpha \Delta x \quad (1)$$

From this equation they derived as

$$\frac{I_0}{I_T} = 1 + \alpha \Delta x \quad (2)$$

$$\alpha \Delta x = 10^A - 1 \quad (3)$$

$$\alpha \propto 10^A - 1 \quad (4)$$

Where A is the absorbance, α is the absorption coefficients, I_0 and I_T are the incident photon intensity and transmitted photon intensity respectively.

Using the equation (4), the absorption coefficients (α) calculated from absorbance spectrum using the modified equation [9]. Here the importance is given only to the linear part of the Tauc plot and approximated the value of absorption coefficients by considering absorption coefficient is proportional to the absorbance [9]. This value of alpha is substituted in the tauc plot equation (5) [10].

$$(\alpha h\nu)^{\frac{1}{n}} = A(h\nu - E_g) \quad (5)$$

Where h is plank's constant, ν is the photon frequency, α is the absorption coefficient, E_g is the band gap and A is proportionality constant. The value of the exponent denotes the nature of the electronic transition, such as $n=1/2$ for direct allowed transition, $n=3/2$ for direct forbidden transition, $n=2$ for indirect allowed transition, $n=3$ for indirect forbidden transition. Generally the allowed transitions dominate the basic absorption process, either $n=1/2$ or $n=2$, for direct and indirect transitions respectively. By plotting and by finding the best fit of the $(\alpha h\nu)^{\frac{1}{n}}$ vs $h\nu$ using $n=1/2$ and $n=2$ gives the correct transition type. Band gap is found from the intersecting point of the curve in the energy axis. Here it is assumed that the optical absorption strength depends on the difference between the photon energy and the band gap.

ZnO has a direct allowed transition [6] and drawn the Tauc plot by substituting $n=1/2$. At low energies the photon energy absorption approached to zero and at higher energies the absorption process saturate and the curve again deviate from linear, which is the characteristic of the Tauc Plot. At the near of the band gap the the absorption gets stronger

and shows a region of linearity in the plot. This linear regions used to extrapolate to the x-axis intercept to find the band gap value.

The Tauc plots of the prepared powder samples are given in the Figure 4 and the band gaps found from the plots are given in the Table 2. It is observed that the band gap has not shown much changes with the concentration of the *Curcuma aromatica Salisb.* solution. This implies that the *Curcuma aromatica Salisb.* has not any remarkable effect on controlling the particle size of the ZnO nanoparticles when ZnO nanoparticles prepared by this method.

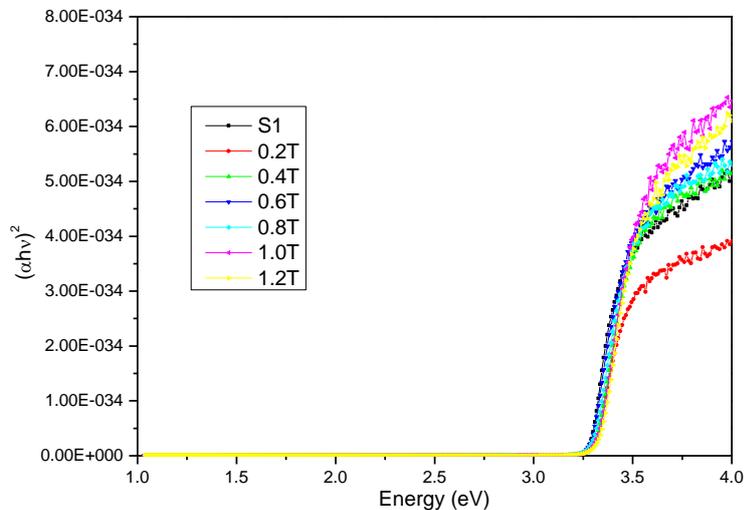


Figure 4. Tauc plots of the prepared ZnO powder samples

Table 3. Band gap and Urbach energy of the eco-dyed ZnO

Sample	Band Gap (eV)	Slope from $\ln(\alpha)$ vs $h\nu$ curve	Intercept	Urbach energy
S1	3.33	10.10142	-31.63335	0.0989
0.2T	3.28	10.15605	-31.4826	0.0984
0.4T	3.28	10.23398	-31.60108	0.0977
0.6T	3.25	10.37584	-31.99135	0.0963
0.8T	3.26	10.55659	-32.58798	0.0947
1.0T	3.28	10.14698	-31.35804	0.0985
1.2T	3.29	10.02314	-31.07025	0.0997

The UV-visible absorption spectra also gives an information about the Urbach tails related to the width of the localized states available in the optical band gap of the ZnO nanoparticles. The band structure in the semiconductor may be damaged due to the disorder in the crystals or may be due to the addition of other extra atoms. Therefore the Urbach energy found below the absorption band edge gives us an idea about the amount of the damage happened to the crystal. For highly imperfect crystal, Urbach energy is large. The generation of absorption edge at the band gap energy is due to the exciton phonon interaction or may be due to the electron phonon interaction. The defects in crystals also absorb light but does not contribute to free electrons instead it recombined or trapped there. This is the curve in the tauc plot after the absorption edge. Urbach energy is the energy that gives the spectral dependence of the absorption coefficients that are examined at photon energies, which are less than the band gap of the material. That is, the formation of localized states with energies at the boundaries of the energy gap which is one of the effects of the structural disorder on the

electronic structure of the material. These defect states traps the excited electrons and prevent the movement of electron to the conduction band when the samples are irradiated with light of a particular wavelength. This absorption tail is called the Urbach tail and is associated with the Urbach energy. The equation for calculating the Urbach energy is

$$\alpha = \alpha_0 + e^{\frac{h\nu}{E_u}} \quad (6)$$

Where α is the absorption coefficient, E_u is the Urbach Energy. The Urbach energy is calculated by plotting $\ln(\alpha)$ vs $h\nu$. Figure 5 shows the Urbach energy plots for all the samples. The slope and intercept of the linear region found by using linear quick fit gadget tool in the Origin Software. The reciprocal of the slope of the linear fit, gives the Urbach energy values and the E_u values are tabulated in Table 3 with band gap energies. There is not seen any trend of changing the disorders as varying the tamarind seed coat tannin concentrations.

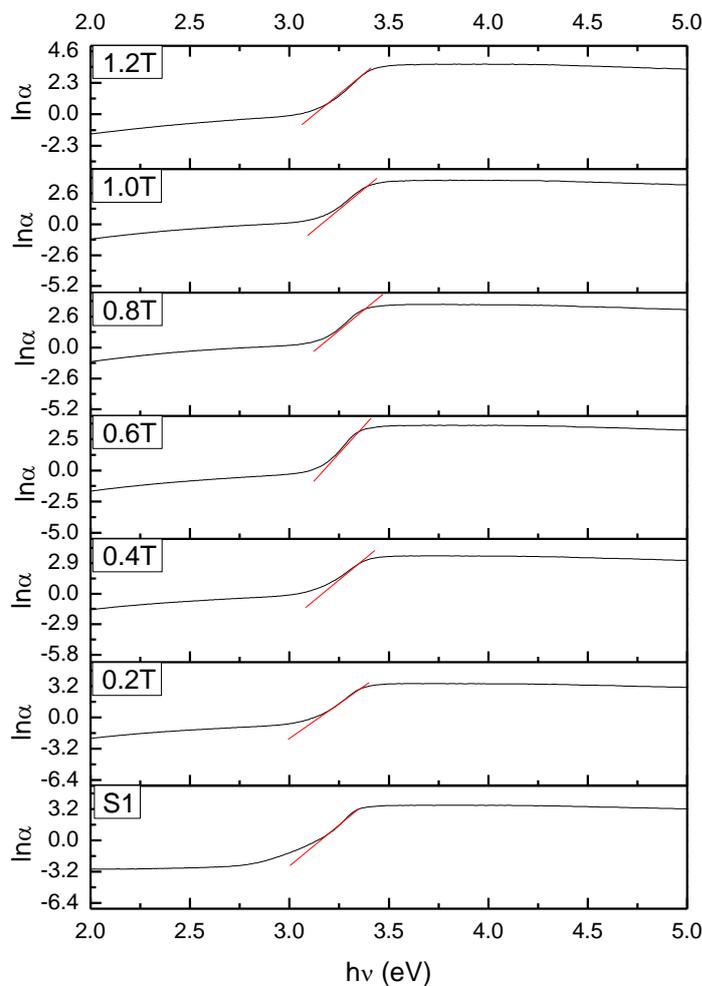


Figure 5. Urbach energy curve for ZnO samples prepared in *Curcuma aromatica Salisb.*

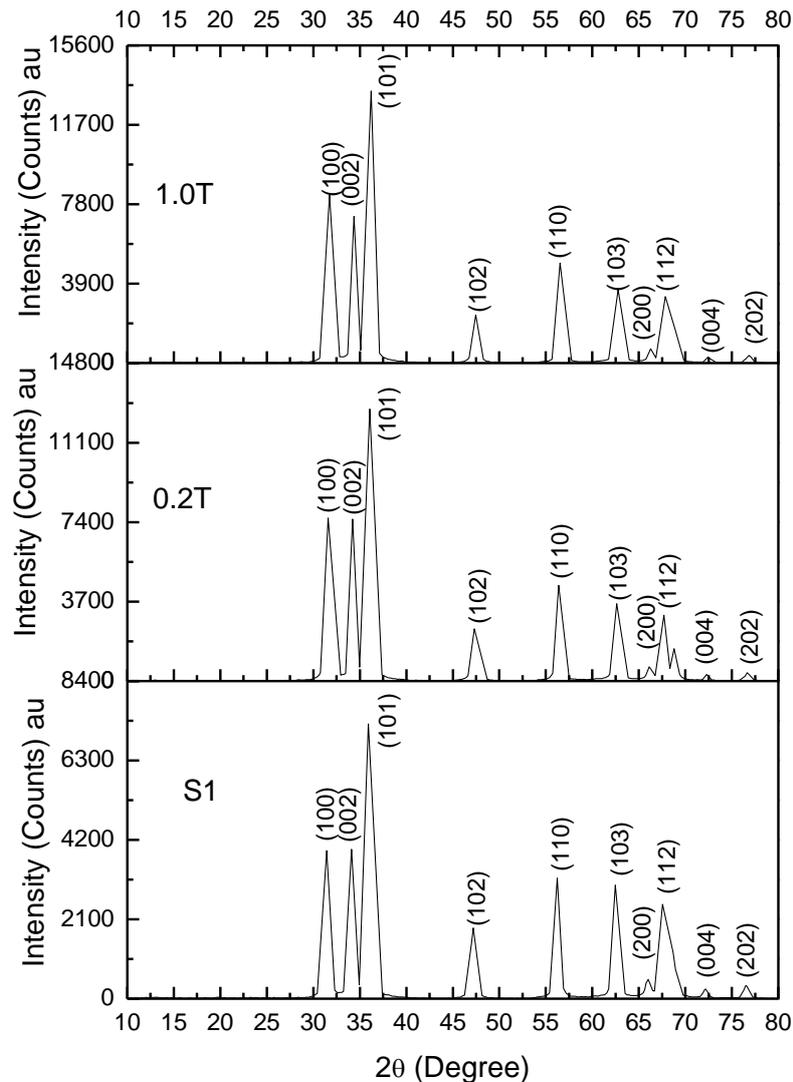


Figure 6. XRD spectra of the prepared ZnO powder samples prepared in *Curcuma aromatica Salisb*.

XRD spectrum shown in Figure 6 has prominent peaks corresponding to the diffraction peaks at $2\theta = 31^\circ, 34^\circ, 36^\circ, 47^\circ, 56^\circ, 62^\circ, 65^\circ, 67^\circ, 72^\circ$ and 76° were indexed with the diffraction planes (100), (002), (101), (102), (110), (103), (200), (112), (004) and (202) which well correlates with the JCPDS card no. 36-1451. The XRD pattern shows that the prepared ZnO has hexagonal wurtzite structure (with $a = b = 3.25 \text{ \AA}$, $c = 5.20 \text{ \AA}$) belonging to the C_{4v} space group (P63mc). The broadening of peak in the XRD pattern clearly implies that small nanocrystals are present in the samples. There is no evidence of bulk form of the materials or any impurity. In the XRD pattern, the (101) diffraction peak is much stronger than other peaks. This indicates that the formed ZnO nanocrystals have a preferential crystallographic (101) orientation. The average crystallite size of prepared sample was calculated by the Debye-Scherrer's formula^[11] i.e.,

$$D = \frac{0.9\lambda}{\beta \cos\theta} \quad (7)$$

where, D is the particle size, λ the wavelength of x-rays ($\lambda = 1.5406 \text{ \AA}$ for Cu $K\alpha$), θ the Bragg angle and β is the full width at half maxima (FWHM). Average particle size (D) of synthesized ZnO nanoparticles were found to be 17 nm using this equation (7).

The induced micro strain in the crystal due to the crystal imperfection and distortion was calculated using the equation ^[12]

$$\epsilon = \frac{\beta}{4 \tan \theta} \quad (8)$$

The strain and particle size can also be found from the Williamson-Hall Plot. Williamson-Hall Plot were drawn using the equation

$$\beta \cos \theta = \frac{k\lambda}{D} + 4 \epsilon \sin \theta \quad (9)$$

Table 4. Geometric parameters of the ZnO nanoparticles of sample S1

2 θ (degree)	Interplanar distance, d (\AA m)	Standard JCPDS, d (\AA m)	hkl	Crystallite Size, D (nm)	Micro strain, ϵ
31.43	2.84396	2.8143	100	16.9	0.007583
34.091	2.62785	2.6033	002	21.5	0.005507
35.921	2.49802	2.4759	101	17.6	0.006394
47.19	1.92444	1.9111	102	17.7	0.004905
56.225	1.63475	1.6247	110	17.8	0.004141
62.488	1.48511	1.4771	103	18.7	0.003567
65.992	1.41449	1.4071	200	13.4	0.004762
67.554	1.38552	1.3781	112	12.9	0.004822
72.158	1.30803	1.3017	004	20.2	0.002918
76.516	1.24402	1.2380	202	17.5	0.003192
Average				17.4	0.004779

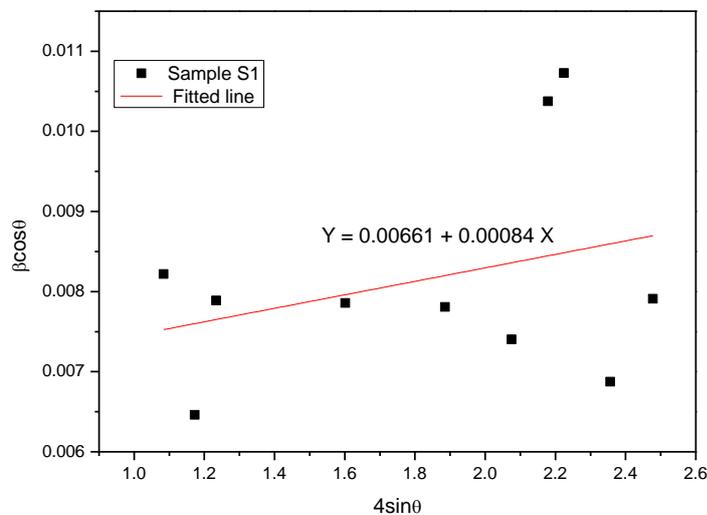


Figure 7. Williamson – Hall plot pristine ZnO sample (S1)

The plots were made by taking $4 \sin \theta$ along x-axis and $\beta \cos \theta$ along y-axis for all the ZnO samples as shown in Figures 8, 9 and 10. From the linear fit, the crystallite size was

estimated from the y intercept and the strain ϵ from the slope of the fit [12]. Geometric parameters of the prepared ZnO nanoparticles are tabulated for the samples S1, Tm0.2 and Tm1.0 are given in the Table 4,5 and 6. Table 7 shows the values obtained from Scherrers formula and from the Williamson-Hall plot. When comparing the tabulated values, it is seen that the size of the crystallites have increased little from 17 nm to 26 nm in the case of Tm0.2 sample and to 21nm in the case of Tm1.0 sample. But, this increase is not evident from the optical absorption analysis. TEM and HRTEM images of the smaple S1 and 1.0T are shown in Figure 10 and 11 respectively. The figures shows that the sizes of the prepared particles are below 50 nm. The interplar spacing measured as 0.22 nm corresponds to the (101) plane.

Table 5. Geometric parameters of the ZnO nanoparticles of sample Tm0.2

2 θ (degree)	Interplanar distance, d (Å m)	Standard JCPDS, d (Å m)	hkl	Crystallite Size, D (nm)	Micro strain, ϵ
31.576	2.83111	2.8143	100	22.5	0.005663
34.227	2.61756	2.6033	002	29.1	0.004053
36.062	2.48859	2.4759	101	22.6	0.004946
47.331	1.91903	1.9111	102	19.2	0.00449
56.375	1.63083	1.6247	110	20.2	0.003623
62.618	1.48255	1.4771	103	19.5	0.003414
66.129	1.41189	1.4071	200	88.9	0.000715
67.685	1.38316	1.3781	112	15.7	0.003965
68.814	1.3632	1.3017	004	15.5	0.00395
72.276	1.30619	1.2380	202	24.1	0.002439
76.67	1.2419	2.8143	100	18.2	0.003073
Average				26.9	0.003667

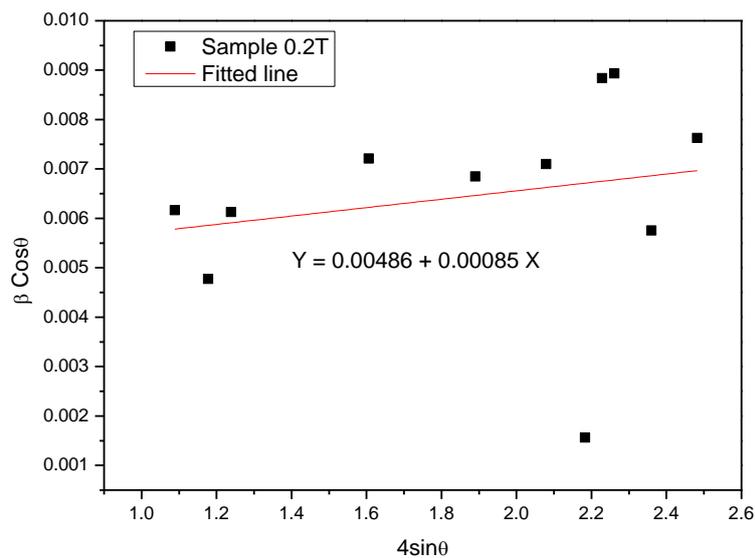


Figure 8. Williamson – Hall plot pristine ZnO sample (0.2T)

Table 6. Geometric parameters of the ZnO nanoparticles of sample 1.0T

2θ (degree)	Interplanar distance, d (Å m)	Standard JCPDS, d (Å m)	hkl	Crystallite Size, D (nm)	Micro strain, ε
31.726	2.81842	2.8143	100	24.6	0.005159
34.376	2.60686	2.6033	002	27.3	0.004288
36.211	2.47857	2.4759	101	23.8	0.004684
47.48	1.91337	1.9111	102	18.2	0.004742
56.527	1.62682	1.6247	110	22.4	0.003262
62.765	1.47917	1.4771	103	19.5	0.003419
66.285	1.40893	1.4071	200	16.5	0.003839
67.839	1.3804	1.3781	112	13.4	0.004633
68.969	1.36052	1.3017	004	17.8	0.003434
72.432	1.30375	1.2380	202	17.4	0.003364
76.832	1.23969	2.8143	100	17.4	0.003196
Average				21.8	0.004402

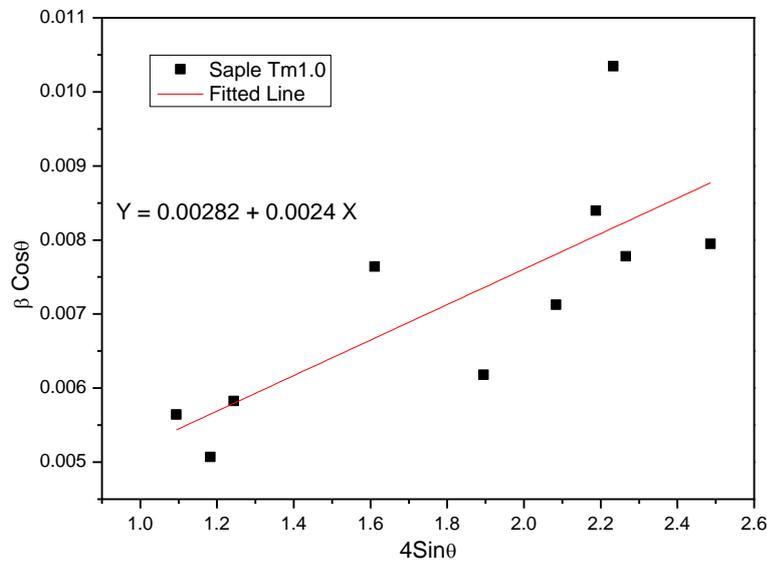


Figure 9. Williamson – Hall plot pristine ZnO sample (Tm1.0)

Table 7. Crystallite size and microstrains of the samples S1, Ta02 and Ta1.2

Sample Name	Average Crystallite Size using Scherrer Formula (nm)	Average crystallite size from W-H plot	Micro strain from eqn.	Micro strain from W-H plot
S1	17.4	18.9	0.0047	0.00080
0.2T	26.9	25.8	0.0036	0.00085
1.0T	21.8	44.3	0.0024	0.00440

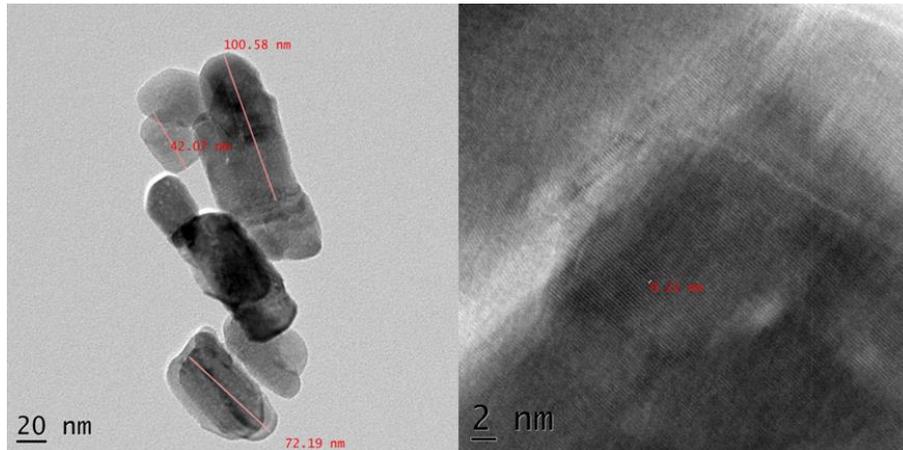


Figure 10. TEM and HRTEM images of the Sample S1

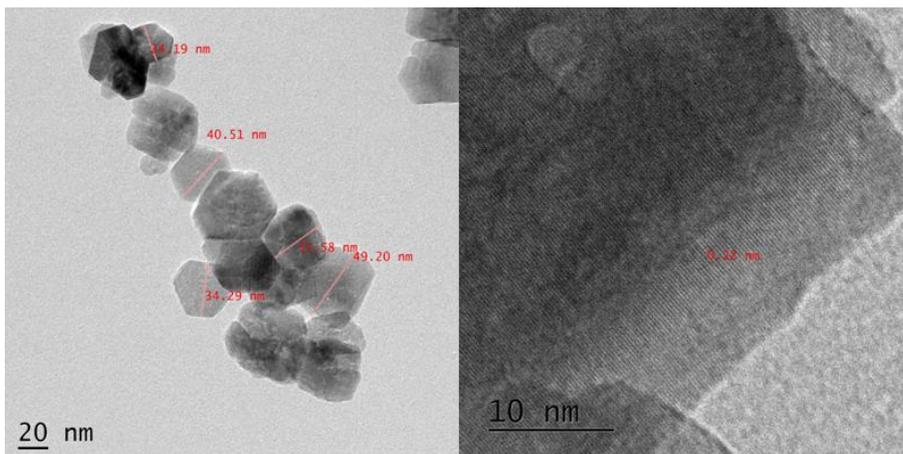


Figure 11. TEM and HRTEM images of the sample 1.0T

CONCLUSIONS

The extracts of *Curcuma aromatica Salisb.* was obtained in deionized water for eco-dyeing ZnO nanoparticles and their FTIR and absorption were studied. The particle size of the prepared ZnO particles were calculated as approximately 17 nm and the particle sizes were seen increased when they prepared in *Curcuma aromatica Salisb.* extract by the proposed method. The UV-visible absorption spectroscopic results shows that the optical behaviour is enhanced and the eco-dyed ZnO can absorb a wide spectrum of light from 400 nm to 650 nm. *Curcuma aromatica Salisb.* extract can be successfully employed as a natural dye to sensitize ZnO nanoparticles without changing their size much.

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