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Synthesis, characterization and activitiesof Zinc oxide nanoparticles using plant extracts

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ABSTRACT

Biological reduction agents are being explored worldwide to minimize the effects of toxic chemicals used in nanoparticle fabrication. The present study states a green approach for the synthesis of zinc oxide nanoparticles employing aqueous stem extract ofAnthocephalus cadamba.Stem extract was used as the biological reduction agent for synthesizing zinc oxide nanoparticles from zinc acetate dihydrate. Synthesis conditions were optimized for maximal and narrow size range synthesis of zinc oxide nanoparticles.The resultant nanopowder was characterized using various analytical techniques, such as UV–Visible spectroscopy, Fourier Transform Infrared spectroscopy, X-ray diffraction and Transmission Electron Microscopy. Nanoparticles were tested for their antioxidant activity of ABTS andHydroxyl radical scavenging activity.

Key Words: Anthocephalus cadamba, Zinc oxide nanoparticles, Green synthesis, Anti-oxidant activity of ABTS, Hydroxyl radical scavenging activity.

1. INTRODUCTION

Anthocephalus cadamba(Family-Rubiaceae) commonly called kadamba enjoys ahallowed position in Ayurveda-an Indian indigenous systemof medicine.It is also named as Kadam. The tree is a medium to large sized deciduous tree attaininga height of 20-40 m and a girth of about 2-2.5 m with cleancylindrical branches and rounded crown. It is frequently foundall over the India on the slopes of evergreen forests up to500 m [1]. Thebark of the plant is reported to possess tonic, bitter, pungent,sweet, acrid, astringent, febrifugal, anti-inflammatory, digestive,carminative, diuretic, expectorant, constipating and antiemeticproperties and is given to treat the fever and inflammationof eyes. The flowers are used as vegetable. The leaves areslightly aromatic with unpleasant taste but the decoction ofleaves good for ulcers, wounds, and metorrhea [2,3]. Additionally,it is useful in the treatment of snake-bite.

Nanotechnology is significant on account of its pre-eminence upon the comprehension, use, and control of matter at magnitudes of a minute scale, akin to approaching atomic levels, with which to manufacture new substances, instruments, and frameworks [4]. The synthesis of nanocrystals is in the limelight in modern nanotechnology. Biosynthesis of nanoparticles by plant extracts is currently under exploitation [5]. Nanotechnology is currently employed as a tool to explore the darkest avenues of medical sciences in several ways like imaging [6], sensing [7], targeted drug delivery [8], gene delivery systems [9], and artificial implants.Zinc oxide (ZnO), a multi-tasking metaloxide, is considered to be one of the best metal oxides that can be used at a nanoscale due to itsunique optical and electrical properties [10]. Nanoparticles can be synthesized either chemically or biologically. But the chemical process for synthesis of Zinc nanoparticles is more elaborate and leaves behind toxic effect that adversely affects the ecosystem.These On the other hand, biological synthesis of Zinc nanoparticles is less time consuming, less costly, and more ecofriendly; therefore, in recent time, scientists are looking forward to the possible biological methods for the synthesis of zinc nanoparticles [11]. In the present study, the green synthesis of Zinc nanoparticles from the *Anthocephalus cadamba*stem extract has been carried out and characterized by UV-Vis spectra, SEM, TEM, and FTIR analysis. The anti-oxidant activity of synthesized ZnO NPs was determined.

2. MATERIALS AND METHODS:

2.1 Materials:

Zinc acetate dihydrate and all other chemicals were purchased from Merck Scientific India Pvt. Ltd., Mumbai.

2.2 Collection of Plant Material:

Fresh stem of the plant *Anthocephalus cadamba* was collected in the Godavari River area near Rajahmundry city, Andhra Pradesh, India

2.3 Collection of the extracts:

Collected plant parts were cleaned with water absorbent paper (wet filter paper). Then it was cut into small pieces, dispensed in 100 ml of sterile distilled water and boiled for one hour at 80ºC. Then the extract was collected in separate conical flasks by standard filtration method.

2.4 Preparation of Extract:

The plant extract was prepared based on the procedure described by *Behzad Shareghi et al*(2016) [12]. 10 g of plant powder was dissolved in 100 ml of distilled water and boiled under constant stirring using a magnetic stirrer for around 3 h. After cooling, the extract was centrifuged twice for 10 min at 4500 rpm and the supernatant was filtered using Watman filter paper and stored at 4ºC for the green synthesis of ZnO nanoparticles.

2.5 Synthesis of ZnO nanoparticles:

Green synthesis of ZnO nano particles was synthesized as per the procedure described by the plant extract was prepared based on the procedure described by *Behzad Shareghi et al* (2016)[12] briefly 0.1, 0.01 and 0.001M aqueous solution of zinc acetate was used as the precursor. The composition of precursor (Zn^{2+} solution) and the plant extract in 9:1 volume ratio were prepared by adding the *Anthocephalus cadamba*extract drop by drop to zinc acetate solution, with constant stirring. The color change in the solution was measured periodically and the formation of ZnO nano particles was determined using UV-Visible spectral studies.

2.6 Characterization of synthesized ZnO nano particles:

2.6.1 UV–Visible spectroscopy:

For UV–Visible spectroscopy, the resultant nano-powder from each of the reactions was re-suspended in equal amount of sterile de-ionized water and spectrum scans were performed using UV–Vis Spectrophotometer Techomp (UV-2301) Analytical Instrument in the wavelength range of 300–800 nm. The absorption values were re-plotted using Hitachi UV solution software version 2.

2.6.2FT-IR spectroscopy:

Fourier transform infrared (FT-IR) spectroscopy helps establish the identity of various phyto-chemical constituents involved in the reduction and stabilization of the nanoparticles. FT-IR spectrum for dried and powdered ZnO NPs was obtained using Perkin Elmer FT-IR Spectrophotometer Frontier using the technique of Attenuated Total Reflectance (ATR) in the range of 4000–500 cm^{-1}.

2.6.3X- ray diffraction (XRD):

The crystallinenature of the synthesized zinc nano particles was studied using XRD study. For the XRD analysis ashed and dried sample of ZnO NPs was used using Bruker- D4 ENDEAVOR (manufacture by Bruker

Corporation) at the wavelength of 1.5406 Å. XRD was performed in the 2θ range of 20–80 degrees at 40 kV and 40 mA with a divergence slit of 10 mm in 2θ/θ continuous scanning mode.

2.6.4 Scanning electron microscope:

The morphology and shape of the zinc oxide nanoparticles were examined using field emission electron microscopy (LEO 1420 VP Compact variable pressure Digital SEM manufacture by Leo Electron Microscopy Ltd).

2.7 Study of antioxidant capacity

2.7.1 ABTS radical activity test

The ABTS method used was based on [14] with slight modifications equal volumes of stock solutions of $ABTS^{+}$ (7 mM) and potassium persulphate (2.45 mM) were mixed and allowed to react for 12–16 h in the dark at room temperature to generate the free radical.Prior to use, this solution was diluted with 60% ethanol to get an absorbance of 0.700 ± 0.020 at 734 nm. An aliquot (5–50µg/ml) of nano particle sample solution was mixed with 1 ml of the working ABTS+ solution and the absorbance was monitored at 734 nm for 6 min using Double Bean UV Visible Spectrophotometer (TECHCOMP – UV 2301, HITACHI 2.2).

% Inhibition = [(AB − AA)/AB] × 100

(Where AB – absorption of blank sample; AA – absorption of tested sample solution).

*The measurements were taken in triplicate.

2.7.1.2 Hydroxyl radical scavenging activity

According to the scavenging activity of the extract on hydroxyl radical was measured according to a previously described method. In 1.5 mL of each diluted extract, 60 µL of $FeCl_3$ (1 mmol/L), 90 µL of 1, 10-phenanthroline (1 mmol/L), 2.4 mL of 0.2 mol/L phosphate buffer, pH 7.8 and 150 µL of H_2O_2 (0.17 mol/L) were added respectively. The mixture was then homogenized and incubated at room temperature for 5 min. The absorbance was read at 560 nm against the blank. The percentage of the hydroxyl radical scavenging activity of each extract was calculated from the equation below:

% hydroxyl radical scavenging activity= [(OD control-OD sample)/OD control] × 100

Where OD is the optical density.

*The measurements were taken in triplicate.

3 RESULTS AND DISCUSSIONS:

3.1 Synthesis of ZnO nanoparticles:

The three different concentrations (0.1, 0.01 and 0.001M) of zinc acetatedehydrate solutions used as the precursor. The composition of precursor and the plant extract in 9:1 volume ratio were prepared by adding the *Anthocephalus cadamba*extract drop by drop to zinc acetate solution, with constant stirring. The colour change in the solution measured periodically represented by **Figure 1**. Based on the yield and time taken for the formation of ZnO nano-particles, 0.01M concentration of metal solution and *Anthocephalus cadamba*as a biological reducing agent selected for further studies. The results were shown in the Table 1.

Figure 1: synthesizes of ZnO NPs from the stem extracts of *Anthocephalus cadamba*

Table 1: Yield of Zn Nano particle:

S.No	Dry weight of ZnO NPs obtained after 6 h of time		
	0.1M	**0.01M**	0.001M
1	11mg	**21mg**	8mg

3.2 Characterization of nanoparticles

In the present study, reduction of zinc ions present in the aqueous solution of zinc acetate during the reaction with the ingredients of *A.cadamba*stem extract has been seen by the UV-Vis spectroscopy ranging from 340 to 800 nm.The maximum absorption was obtained at 368nm (Figure 2).The bioreduction of zinc acetate ions in solution was monitored by periodic sampling of aliquots (0.1 mL) of aqueous component and measuring UV-Vis spectra of the solution. UV-visible spectra show no evidence of absorption in the range of 400–800 nm for the plant extractand the plant extract solution exposed to zinc acetate ions shows a distinct absorption at around 368nm (Figure 2) which corresponds to surface plasmon

resonance (SPR) of zinc nanoparticles established at 370nm in previous studies [13].

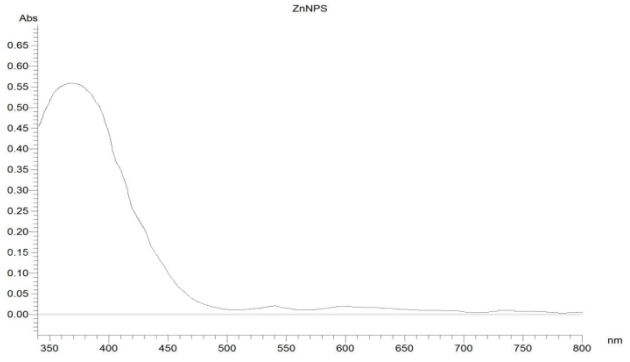


Figure 2:UV-visible scanning spectra of synthesized ZnNPs

FTIR spectra of green synthesis ZnO NPs (Figure 3) have shownabsorption band at 3707 cm^{-1} indicates the presence of Amide N-H Stretch or OH groups in alcohols. Two medium bands were observed at 3426 and 33573707 cm^{-1}confirms primary amine groups. Strong bond at 2998 and 2943 cm-1 indicates C-H stretching vibrations. Strongbond at 1731 cm^{-1} represents C=O stretch vibrations in carbonyl compounds. Medium band at 1367 representingC-Nstretchin amines was also observed in IR spectra. C=C aromatic stretching was noticed at 1464cm^{-1}. The shift observed in FT-IR spectra of ZnO NPs after bioreduction band indicate the participation of polyols, terpenoids,and proteins having functional groups of amines, alcohols, ketones, and carboxylic acid inbioreduction reactions.Terpenoids are poorly water-soluble and hence may not be amongprime moieties involved in the bioreduction reaction. However, proteins seem to exhibit littleimportance in biosynthesis of nanoparticles as reported earlier [14].Therefore, water-solublephenolic acid and flavonoid compounds are believed to play a major role in bioreductionreaction. But, the possible mechanism is still unclear and needs further investigation.

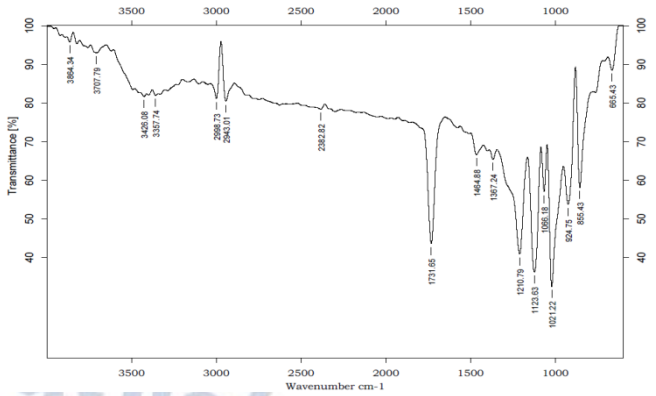


Figure 3: FT-IR spectra of synthesized ZnO NPs

The crystalline size, shape and surface morphology of the ZnO nanoparticles aredetermined by using SEM as shown in the figure 4. The micrograph images of ZnOnanoparticles prove that they are in nanoscale range and have a uniform distribution withspherical shape and less aggregation is observed. The average particle size was found to be 167nm. The results depict polycrystalline with porous nature of ZnO NPs.

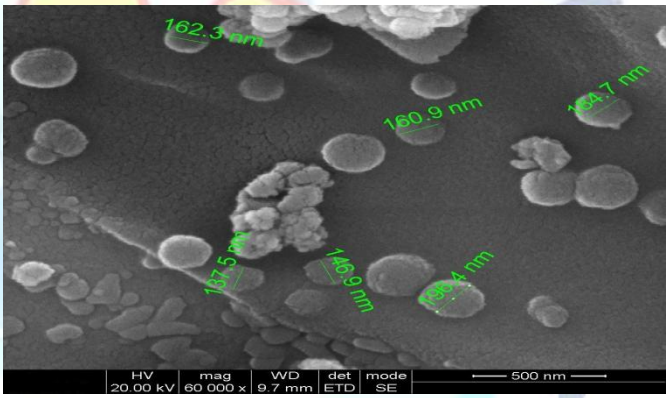
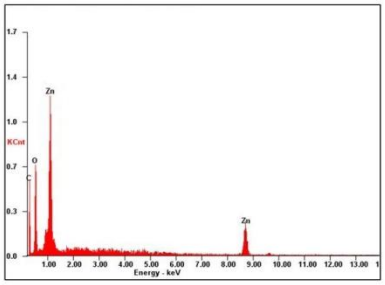


Figure 4: SEM images of synthesized ZnNPs

The EDX pattern ofthe ZnO nanoparticles is shown in Fig. 4. The C signal observed in EDX spectra is from the carbon film coated on the supportformvar. A characteristic signal corresponding to Zn metal was observed in the spectra. No other signals were detected within thedetection limits of EDS which confirm the purity of the ZnOnanoparticles. The % of Zn content in the crystalline particle was found to be 25.29%.



Element	Wt%	At%
CK	38.37	53.96
OK	26.34	37.91
ZnK	25.29	8.13
Matrix	Correction	ZAF

Figure 4: EDX results of synthesized ZnO NPs

3.3.1 ABTS radical activity test

In the present investigation, the commonly accepted assay ABTS was used for the evaluation of antioxidant activity of plant root extracts the results of these analyses are given in **Table 2.** The relative antioxidant ability to scavenge the radical ABTS+ has been studied for ascorbic acid, root based ZnO Nps and aqueous root extracts and the antioxidant power was measured by studying decolorization. The ABTS values for root based Nps ranged from. 4.79 to 86.88 %, the results are comparable to the ascorbic acid and for aqueous root extracts and IC50 values were calculated. From the results it is to be noted that Root based ZnO NPs have IC50 value of 27.60µg/ml, it is nearer to the value ofAscorbic acid i.e 25.48 and the IC50 value of Aqueous Root extract is 41.76. The antioxidant effect of Zno nanoparticles was stronger than other synthetic commercial standard used. The ABTS radical cation decolorization assay can measure the relative antioxidant ability to scavenge the radical ABTS as compared with BHT, and is an excellent tool for determining the antioxidant capacity of hydrogen-donating antioxidants. The blue and green ABTS radical cation be generated prior to adding up antioxidant containing samples prevents interference, which stable absorbance was achieved, by adding the ethanolic extract of *Anthocephalus cadamba* and the scavenging ability measured in terms of discolorization [14]

Table 2: ABTS radical activity test of Root extract

S No	Concentration in µM	Ascorbic acid		Root based ZnO NPs		Aqueous Root extract	
		Absorbance	% ABTS Activity	Absorbance	% ABTS Activity	Absorbance	% ABTS Activity
1	5	0.639	7.20	0.678	4.79	0.694	1.97
2	10	0.564	16.40	0.602	15.28	0.653	8.20
3	15	0.501	29.45	0.536	24.76	0.603	14.95
4	20	0.428	40.49	0.458	35.46	0.549	22.78
5	25	0.320	55.01	0.368	47.95	0.50	29.62
6	30	0.270	62.62	0.302	57.93	0.440	38.18
7	40	0.133	81.43	0.155	77.87	0.374	47.53
8	50	0.068	90.46	0.098	86.84	0.294	58.54

3.3.2 Hydroxyl radical scavenging activity

The potential of an ethanolic root extract of *Sphagneticola trilobata* to inhibit hydroxyl-radical-mediated deoxyribose damage was assessed at a concentration of 5 µM to 100µM. The sample exhibited minimum activity of 3.02 % at 5 µM and maximum activity of 93.15 at 100 µM, showing that the hydroxyl radical scavenging activity occurred in a dose-dependent manner Table 3. The results indicate the scavenging potential against hydroxyl radicals. Superoxides are produced from molecular oxygen by oxidative enzymes as well as via nonenzymatic reactions such as auto-oxidation by catecholamines. Superoxide anions play an important role in the formation of other reactive oxygen species such as hydrogen peroxide, hydroxyl radical and singlet oxygen, which induce oxidative damage in lipids, protein and DNA [15]. The superoxide scavenging activity of *Sphagneticola trilobata* was investigated, because the extract has the potential to scavenge superoxide anions.

Table 3: Hydroxyl radical scavenging activity

S No	Concentration in µM	Ascorbic acid		Root based ZnO NPs		Aqueous Root extract	
		Absorbance	% Activity	Absorbance	% Activity	Absorbance	% Activity
1	5	0.830	4.22	0.856	3.06	0.849	1.94
2	10	0.795	9.34	0.798	7.83	0.785	7.88
3	15	0.729	17.24	0.731	15.39	0.749	13.14
4	20	0.660	23.64	0.686	20.49	0.716	18.06

5	25	0.588	31.13	0.606	29.89	0.646	23.89
6	30	0.519	40.53	0.559	36.29	0.603	28.90
7	35	0.429	50.48	0.489	44.15	0.563	33.43
8	40	0.346	59.81	0.363	57.71	0.501	40.66
9	50	0.241	72.09	0.271	68.52	0.428	49.06
10	75	0.116	86.69	0.170	80.50	0.376	55.42
11	100	0.030	96.79	0.063	93.15	0.308	64.45

4. CONCLUSION:

Green synthesis of circular shaped ZnNPs of about 67nm has beenachieved using stem extract of *Anthocephalus cadamba*, thus bringing intolight yet another use of the plant besides its usual utilities.The water-soluble phenolic acid and flavonoid compounds were believed to play amajor role in bioreduction reaction confirmed by FT-IR. UV spectrum identified peaks werelocated in the range of the blue-violet spectrum had maximum centered 368nm. Antioxidant activity of ABTS and Hydroxyl radical scavenging activity of ZnO NPs increased with increase of concentrations of ZnO nanoparticles.

Conflict of interest statement

Authors declare that they do not have any conflict of interest.

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