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Synthesis and characterization of zinc oxide nanoparticles from aqueous extract of elephant grass (*Pennisetum purpureum*)

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Abstract

Objective: To synthesise and characterise *Pennisetum purpureum* zinc oxide nanoparticles from aqueous extracts of elephant grass (*Pennisetum purpureum*).

Methodology: Zinc oxide nanoparticles were synthesised with aqueous extract of elephant grass (*Pennisetum purpureum*) *and zinc acetate dihydrate.* It was characterized by using various dispersion methods such as UV-visible spectrophotometry, Transmission Electron Microscopy (TEM), Fourier Transform Infrared (FTIR) Spectroscopy and X-ray diffraction (XRD) to determine the morphology, crystallographic structure, chemical composition, physical properties and crystalline size of the nanoparticles.

Results: The results showed that Zinc oxide nanoparticles were synthesized and characterized using various dispersion methods. UV–Vis spectra showed typical absorption peaks around 370nm *(Pennisetum purpureum).* FTIR revealed the presence of biomolecules and functional groups (C-O, O-H, CH, C≡C, C=C, N-O) that performs various functions like stabilizers, capping and coating agents in nanoparticle synthesis. XRD revealed the crystallographic structure, chemical composition, physical properties and crystalline size of the nanoparticles. The average crystalline size of zinc oxide nanoparticles was calculated using Debye–Scherer equation and the average size was 23.37nm.

Conclusion: In this study, simple and green method for the synthesis of zinc oxide nanoparticles using aqueous extract of elephant grass (*Pennisetum purpureum*) and characterization using various dispersion methods was described. The formation of zinc oxide nanoparticle was confirmed by UV-Visible spectroscopy. The FTIR spectrum showed that the phytochemicals found in the plant extract was incorporated into the zinc oxide nanoparticle as capping and stabilizing agents. XRD results revealed the average size of 23.37nm (*Pennisetum purpureum* ZnO) and Transmission Electron microscopy (TEM) confirmed the morphology to be spherical.

Keywords: *Pennisetum purpureum*; Zinc oxide; Nanoparticles; Aqueous extract

1. Introduction

Nanotechnology centers on synthesis of particle with sizes ranging from 1-100 nm. Nanoparticles of various metals including that of silver, gold, copper, zinc, titanium, cadmium and iron have been synthesized (Oberdörster et al., 2005)

The use of plant extracts for the synthesis of nanoparticles is on the rise because of metabolites and phytochemicals present in plants which hasten the synthesis and stabilizes the nanoparticle in solution (Franke et al., 2010).

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Elephant grass (*Pennisetum purpureum* Schum.) is a monocot belonging to the family Poaceae (grass family). It is a common weed found in Nigeria and known to be rich in metabolites such as linolenic, oleic acid, tannins, alkaloids, flavonoids, saponins, cyanogenic glycosides and oxalate. Most of these compounds are reported as useful component in nanoparticle synthesis and perform various functions like stabilizers, capping agent and coating agents in nanoparticle synthesis (Dong *et al.,* 2016). Elephant grass (*Pennisetum purpureum* Schum.) has been exploited in phytoremediation studies (Alikasturi et al., 2020).

2. Material and methods

2.1. Collection, Identification and Preparation of Plant Materials

Fresh leaves of Elephant grass (*Pennisetum purpureum)* were harvested from University of Port Harcourt, Rivers State and identified by Dr. Wisdom Barade (WNB85), a plant taxonomist. The voucher number (317) was obtained from Kenule Beeson Polytechnic, Bori. Rivers State Herbarium. They were washed thoroughly in tap water, shade-dried and homogenized to fine powder and stored in air tight bottle.

2.2. Preparation of Aqueous Extract

100 g of the leave powder was weighed and soaked in 500 ml of distilled water in a conical flask. This was covered, shaken every 30 min for 6 h and then allowed to stand for about 24h. The solution was subsequently shaken and filtered using muslin cloth and refiltered using Whatman number 1 filter paper.

2.3. Synthesis of zinc oxide nanoparticles (green synthesis approach)

2.3.1. Pennisetum purpureum Zinc oxide nanoparticles

The zinc oxide nanoparticles were biosynthesized using the modified method of Sabir et al. (2014). Zinc acetate dihydrate was used as the precursor and *Pennisetum purpureum* aqueous extract serves as the reducing agent. The zinc oxide nanoparticles were biosynthesized using aqueous extract of elephant grass (*Pennisetum purpureum)*. 50 ml aqueous extract was added to 50ml of 0.1M zinc acetate dihydrate ($Zn(CH_3COO)_2.2H_20$) solution in a beaker. The pH was adjusted to pH 12 by addition of 0.2 M NaOH. A pale white aqueous solution was formed. The mixture was stirred for 2 h using a magnetic stirrer and kept in a water bath at 50°C. The formation of a creamy precipitate indicates the presence of zinc oxide nanoparticles. The colloidal suspension was centrifuged at 10000 rpm for 15 min and the supernatant discarded. Pellet was collected and washed with deionized water, followed by ethanol to make it free from impurities then oven dried overnight at 60°C. Pale white powder of zinc oxide nanoparticles was obtained.

2.3.2. Characterization of zinc oxide nanoparticles

The synthesized nanoparticles were characterized by different dispersion techniques. For the UV–Vis analysis, 1 ml of the suspension was collected from the purified sample at the end of the reaction and was sonicated at 4000 rpm for 15 min. The UV–Vis spectra were recorded over the 200–800 nm range. FTIR analysis of the dried zinc oxide nanoparticles was carried out by the KBr pellet method and the presence of the various vibrational modes in the synthesized nanoparticles was investigated. The phase structure and material identification of zinc oxide nanoparticles was studied by X-ray diffractometer. The Transmission electron microscope (FEI–Tecnai G2 20 S-TWIN Germany) (TEM) technique was employed to visualize the size and morphology of the particles.

3. Results and discussion

3.1. UV–Visible spectroscopy

UV-VIS absorption spectroscopy is a widely used technique to examine the optical properties of nanosized particles (Manjunatha et al., 2019). Surface Plasmon Resonance (SPR) was responsible for the unique optical properties of zinc oxide nanoparticles by factors such as particle size, particle shape, their distance from each other (concentration) and refractive index of the changes in the surrounding environment (Salem, 2021). The surface plasmon resonance (SPR) of zinc oxide nanoparticles is between 310–380 nm (Agarwal & Shanmugam, 2020). UV–Visible absorption spectrum of synthesized nanoparticles is shown in Figure 1. The distinct peak centered around 370 nm is specific for zinc oxide nanoparticles (Dangana et al., 2023), which is due to their large excitation binding energy at room temperature (Bhuyan et al., 2015). The colour change was due to the reduction of zinc ions. Metallic nanoparticles scatter and absorb light at certain wavelengths due to the resonant collective excitations of charge density at the interface between a conductor and an insulator known as surface plasmon resonance. Metal nanoparticles have electrons which give surface plasmon resonance absorption band due to the combined vibration of free electrons on its surface in resonance with light wave. In this study, the absorption band of zinc oxide nanoparticles was observed at 370 nm (Fig. 1).

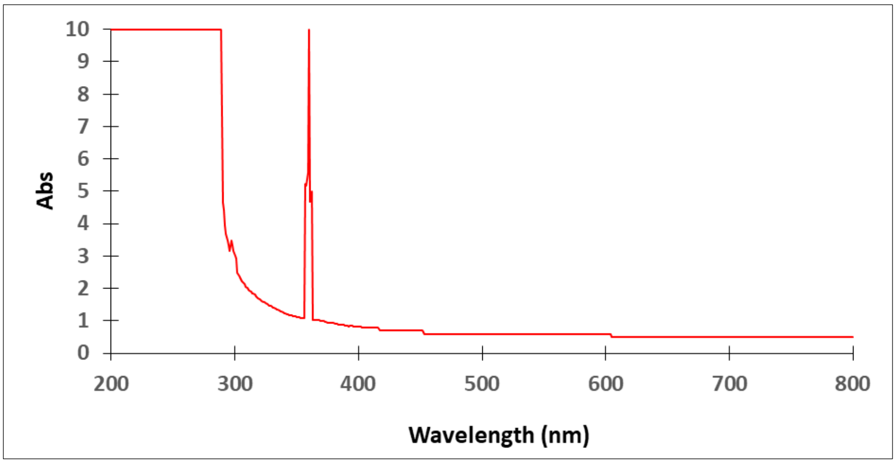

Figure 1 UV-Vis absorption spectrum of ZnO nanoparticles synthesised from *Pennisetum purpureum* aqueous extract

3.2. Fourier Transform Infrared Spectroscopic (FTIR) analysis

Infrared study was carried out in order to ascertain the purity and nature of the nanoparticles and also the presence of biomolecules. The FTIR spectrum of *Pennisetum purpureum* synthesized zinc oxide nanoparticles in figure 2 showed absorption peaks at 3201 cm^{-1} (NH), 2117 cm^{-1} (C≡C), 1543.1cm^{-1} (N-O), 1401.5 cm^{-1} (N=O, $-CH_3$) and 1017.6 cm^{-1} (C-O, O-H). This indicates the presence of biomolecules and functional groups in the plant (elephant grass) that can interact with the zinc surface and aid in the stabilization of zinc oxide nanoparticles.

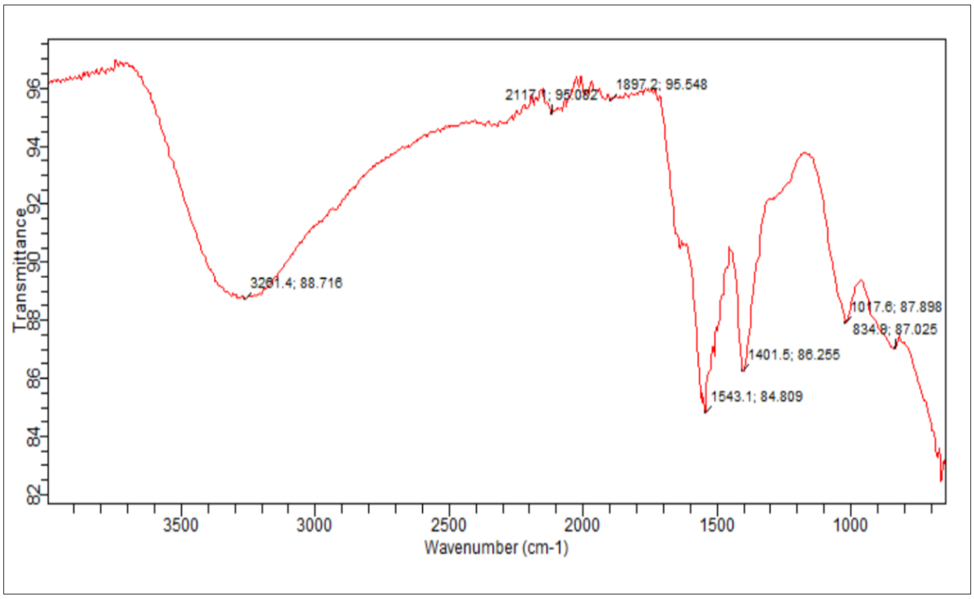

Figure 2 FTIR of *Pennisetum purpureum* zinc oxide nanoparticles

Table 1 FTIR Analysis of *Pennisetum purpureum* zinc oxide nanoparticles

Characteristic Absorption (cm^{-1}) Peak value	Bond	Functional group
1017.6	C-O	Ether, Ester
	O-H	Alcohol
1401.5	N=O	Nitro
	$-CH_3$	Alkane
1543.1	N-O	Nitro
2117	C≡C	Alkyne
3201	NH	Amine and Amide

3.3. X-ray diffraction analysis

XRD results revealed crystallographic structure, chemical composition, physical properties and crystalline size of the synthesised zinc oxide nanoparticles.

Table 2 XRD result for synthesised *Pennisetum purpureum* zinc oxide nanoparticles

Peak position 2θ(^{0})	FWHM B size (^{0})	Dp (nm)	Dp Average (nm)
28.56	0.42	20.40	23.37
31.06	0.23	37.47	
31.80	0.41	21.06	
32.68	0.31	27.91	
34.50	0.24	35.23	
36.33	0.44	19.86	
47.59	0.69	13.15	
56.55	0.56	16.84	
62.86	0.56	17.38	
68.02	0.59	16.98	

The average size of zinc oxide nanoparticles was calculated using the Debye–Scherer equation, where ω is the X-ray wavelength coming from Cu-Kα (1.540560 Å), β is the full width at half maxima of the diffraction peak in radians, θ is the Braggs' angle in degrees, and K is the shape factor and its value is equal to 0.94 (Scherrer's constant)

$$\left(\frac{D = K\omega}{\beta \cos\theta}\right)$$

$$D = \frac{0.94\lambda}{\beta \cos\theta},$$

XRD analyses revealed the average size of (23.37 nm) for *Pennisetum purpureum* zinc oxide nanoparticles. Details of XRD results are given in Table 2-4 and figure 3. The particle size of the synthesized zinc oxide nanoparticles was in close agreement with the previous findings of Shabnam et al., (2019) who synthesised zinc oxide nanoparticles using *Laurus nobilis* leaves aqueous extract and two different zinc salts (zinc acetate and zinc nitrate) as precursors, the XRD analysis revealed the average size of (21.49, 25.26 nm).

Table 3 Details of XRD peak position (2θ) for synthesized *Pennisetum purpureum* zinc oxide nanoparticles

Peak position 2θ(0)	Phase Name	CHEMICAL FORMULA
28.56	quartz HP	Si O2
31.06	Unknown	
31.80	Zincite, Osumilite:	Zn O, K - Na - Ca -Si-Al-Mg-Fe3+
32.68	Unknown	
34.50	Zincite, Clinochlore:	Zn O, Mg , Fe2+, Al2Si3O10(OH)8
36.33	Zincite, Clinochlore:	Zn O, Mg , Fe2+, Al2Si3O10(OH)8
47.59	Zincite, Clinochlore:	Zn O, Mg , Fe2+, Al2Si3O10(OH)8
56.55	Zincite,	Zn O
62.86	Zincite, quartz HP	Zn O, Si O2, (Mg, Fe)
68.02	Zincite, quartz HP	Zn O, Si O2, Ca H P

Table 4 Quantitative results of XRD showing weight fraction (%) of *Pennisetum purpureum* zinc oxide nanoparticles

Phase name	Weight fraction (%)	Chemical formula
Zincite, syn	63.2	ZnO
Quartz HP, syn	5.0	SiO_2
Brushite	4.76	$CaHPO_4\ 2H_2O$
Osumilite	6.66	K-Na-Ca-Mg-Fe-Al-Si-O
Clinochlore	20.4	Mg , Fe^{2+}, $Al_2Si_3O_{10}(OH)_8$

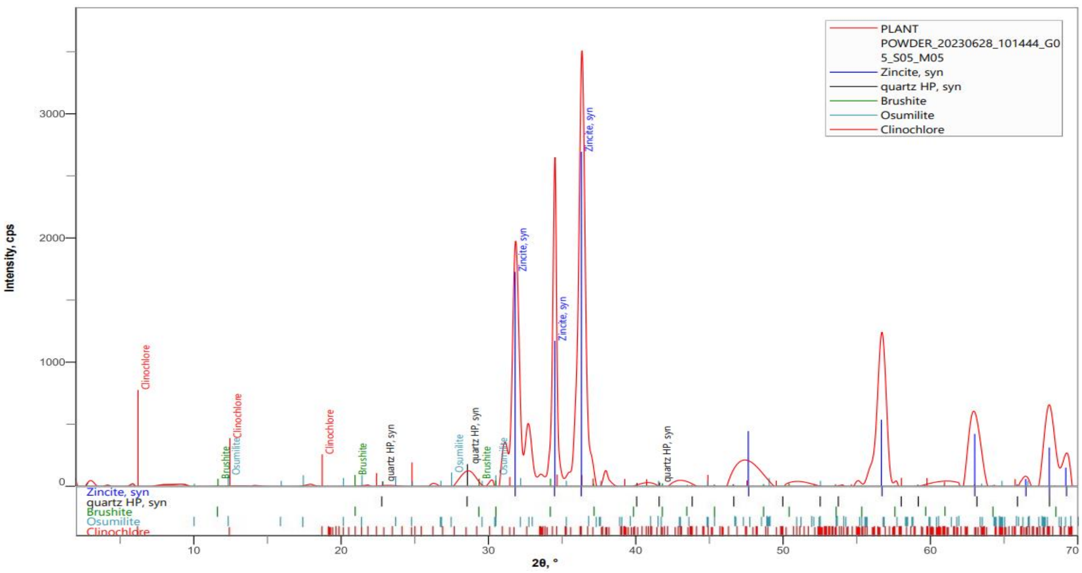

Figure 3 XRD spectrum of *Pennisetum purpureum* zinc oxide nanoparticles

3.4. Transmission Electron Microscopy

Transmission Electron Microscopy (TEM) is a technique that uses an electron beam to image a nanoparticle sample, providing much higher resolution than light-based imaging techniques. Transmission Electron Microscopy is the preferred method to directly measure nanoparticle size, grain size, size distribution, and morphology.

Figure 4-5 showed that *Pennisetum purpureum* zinc oxide nanoparticle is spherical in shape and the sizes ranges from 0.88 to 23.80nm.

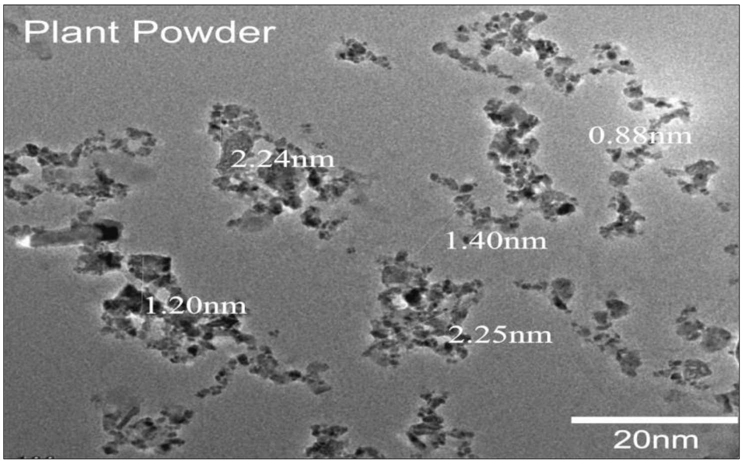

Figure 4 TEM 20nm morphological result of *Pennisetum purpureum* zinc oxide nanoparticles

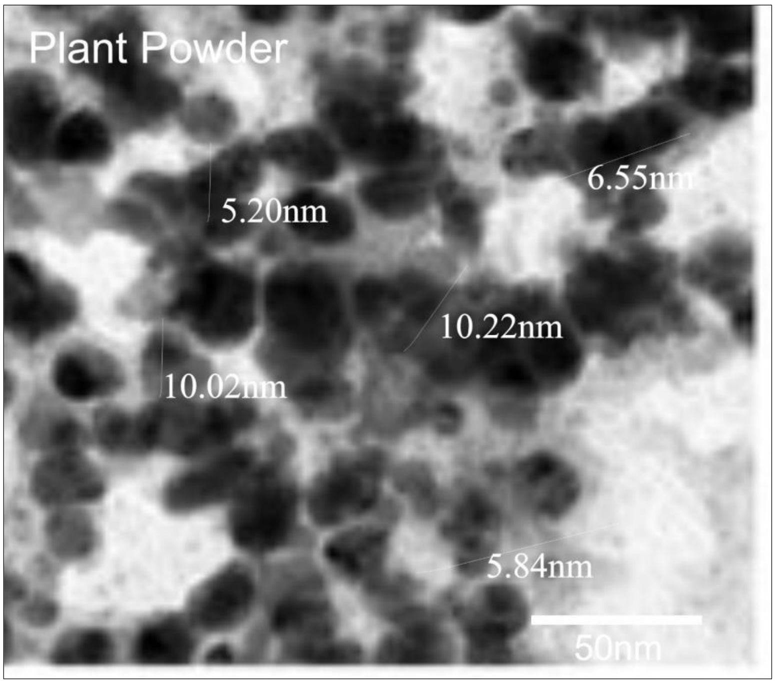

Figure 5 TEM 50nm morphological result of *Pennisetum purpureum* zinc oxide nanoparticles

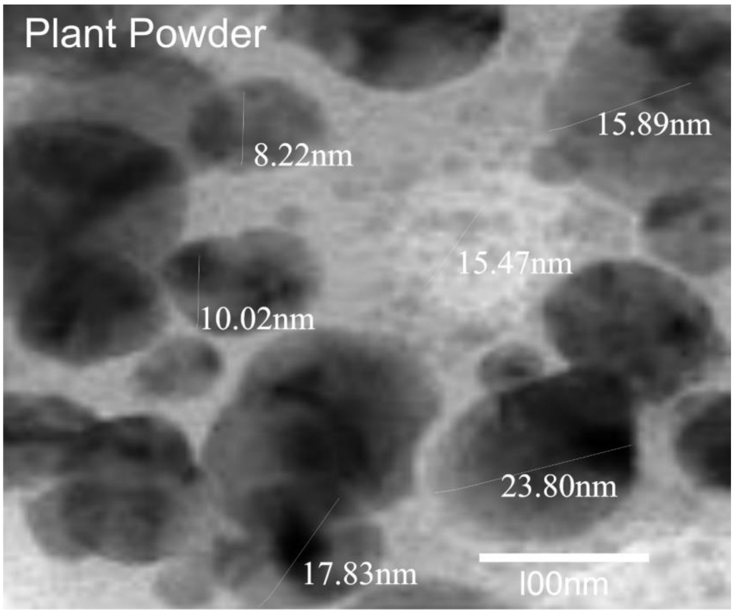

Figure 5 TEM 100nm morphological result of *Pennisetum purpureum* zinc oxide nanoparticles

4. Conclusion

In this study zinc oxide nanoparticle was synthesized by aqueous extract of *Pennisetum purpureum* and zinc acetate dihydrate. UV–Visible spectrum showed a distinct peak around 370 nm, which is specific for zinc oxide nanoparticles. The XRD results confirmed the average size of zinc oxide nanoparticle synthesized to be 23.37 nm, exhibiting spherical structures respectively which were confirmed by XRD and TEM analyses. FTIR studies clearly showed the presence of biomolecules and functional groups in the plant (*Pennisetum purpureum*).

Our results confirmed the potential of *Pennisetum purpureum* for the synthesis of zinc oxide nanoparticles in a simple, fast and ecofriendly way.

Compliance with ethical standards

Disclosure of conflict of interest

No conflict of interest to be disclosed.

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