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RESEARCH ARTICLE

Green Synthesis of ZnO NPs and CdO-ZnO Nanocomposites using aqueous Extract of Water Hyacinth (*Eichhornia crassipes*) Characterization, Structural and Nano-fertilizer using Application

Munusamy Kalaivani[1]*, Subramaniyan Ravi[2]

1 Research scholar, Department of Physics, Faculty of Science, Annamalai University, Chidambaram, Tamil Nadu, India
2 Associate Professor, Department of Engineering Physics, Faculty of Science, Annamalai University, Chidambaram, Tamil Nadu, India

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* **Corresponding author**.

kalaimpsk1512@gmail.com

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Abstract

Objectives: To evaluate structural and morphological character of Zinc oxide and Cadmium doped zinc oxide nanoparticles and its nano-fertilizer property also measured. **Methods:** ZnO and CdO-ZnO nanoparticles synthesized by water hyacinth (*Eichhornia crassipes*), Aqueous extract of water hyacinth (*E. crassipes*) acts as a reducing and capping agent for the synthesis of ZnO and CdO-ZnO nanoparticles. The product was characterized by X-ray diffraction (XRD), Fourier transform infrared spectroscopy, Scanning electron microscopy (SEM) with energy dispersive X-ray analysis (EDX) and UV-Visible spectroscopy to ascertain its formation. With application of Nano fertilizer to low marginal soils for cultivation of cassava, the macronutrients and micronutrients enhanced by Nano fertilizer microbial inoculant that transforms roots development, photosynthetic rate, replenished extracted soil nutrients by cassava harvest and improved nitrogen use efficiency. **Findings:** The Greener method is very useful environmentally to reduce the hazardous solvent and some other chemical reagents. This study has proved the uilization of water hyacinth (*E. crassipes*) extract, as a good reducing agent, for the above nanoparticles synthesis. With the application of Nano fertilizer to low marginal soils for cultivation of cassava, the macronutrients and micronutrients are enhanced by nano fertilizer microbial inoculants that transforms roots development, photosynthetic rate, replenished extracted soil nutrients by cassava harvest and improved nitrogen use efficiency. **Novelty:** The present work authenticated that Nano fertilizer applied cassava production soil provides interactions which produces stability and resilience of soil with respect to functional characteristics. However, the effect of each green manure on cassava plant height, number of branches and yield depends on its chemical composition. ZnO nanoparticles dispersed in Nano fertilizer to cassava plant 0-10 month in experimental trail plant shows significant increase in height branches of leaves and tuber yield for and treatment- 1(ZnO NPs) and cassava treatment-2 (ZnO and CdO-ZnO) , treatment 3 (Chemical fertilizer) and treatment -4 control by 23.91%, 28.91% ,18.67% and 14.58 % respectively, when compared to the control. Applications of Nano fertilizer ZnO NPs and CdO-ZnO dispersed in applied to the cassava plant can significantly enhance its growth and yield performance. These findings make it useful to produce new Nano fertilizer in various metal and metal dopped nanoparticles.

Keywords: Green synthesis; Water hyacinth plant; Nano fertilizer

1 Introduction

A nanofertilizer is any product that is made with nanoparticles or uses nanotechnology to improve Nutrient efficiency [1]. Zinc Oxide is an n-type semiconductor with a wide and direct band gap around 4.09 eV [2] at room temperature depending on the synthesis conditions [3–5]. Nanoparticles exhibit atom-like manners due to lofty surface energy resulting from high and large specific surface area [6], high fraction of surface atoms and wide gap between valence and conduction band when divided to near atomic size [7–9] ZnO is one of the important group II–VI semiconducting materials and it is also applied [10] for making light emitting diodes, gas sensors, field effect transistors, photodetectors, ultraviolet lasers, solar cells and so on due to its inherent properties [11]. Additionally, metal nanoparticles have a surface Plasmon resonance absorption in the UV–visible region. It is non-toxic, self-cleansing , compatible with skin, antimicrobial, dermatological catalysts. Therefore, control of the morphology and size of particulate inorganic materials has received increased and is used as an UV-blocker in sunscreens and many biomedical applications [12]. The depend on inexpensive organic sources of nutrients results from the fact that farmers rarely utilise chemical fertiliser because of its scarcity and high cost. These factors make research on improving the efficacy of organic manures and determining the best rate of application necessary. Nanofertilizers that are used as soil inoculants multiply, take part in nutrient cycling, and increase crop productivity. Cassava has been provided a biotechnology makeover, and numerous studies are actively using it to improve its genetics. Low yields per unit area, a low cassava population, and weed growth on farms were cited as factors for the low adoption rate. Farmers are therefore asking for more cassava to be planted per hill or less or no chemical pesticides to be used during production. According to research by Ramanandam et al. [13], the generation of bioactive compounds with actions similar to growth regulators as well as nitrogen fixation by nanofertilizers led to increased dry matter production. The increased dry matter production is related to the cumulative impact of the growth characteristics' progressive increases, namely, plant height and the number of branches per plant's leaves per branch [14]. Herbicides are used all over the world, however cassava is typically weeded by hand (hoe) 2-3 times during the first 3-4 months. The excitation (nanofertilizer application) to the cassava agriculture soil system is considered for diagnostic testing of "nanofertilizer rhizosphere holistic soil function" due to the functional characteristics of the impacts on rhizosphere. Keeping in view the benefits Tapioca (Manihotes culenta Crantz) plant used the nano fertilizer it offers over the other conventional treatments methods a study was designed to synthesize ZnO nanoparticles and CdO-ZnO nanocomposites through greener route using aqueous leaf extract of Water Hyacinth (*E. crassipes*). To characterize and to apply for the first time as fertilizer control, water $T_{1,}$ ZnO NPs T_2 and Cd/ZnO NPs T_3 in effect of fertilizer medium. Investigation of therapeutic applications of zinc oxide nanoparticles and CdO-

ZnO nano composites was also the objectives of the present study.

2 Methodology

2.1 Materials

Zinc nitrate and cadmium nitrate was purchased from Sigma–Aldrich Chemicals. All the chemicals used in this study were analytical grade. Water hyacinth leaves were obtained from the Chinnanellikollai village, Cuddalore (dt) Tamil Nadu, India. Distilled water was used to conduct all the experiments.

2.2 Sample Preparation of plant extract

The leaves were cleaned as necessary, washed in distilled water and running water to eliminate dust and other contaminants, and then allowed to air dry for seven days at room temperature with no dust present. Dried leaves were formed powder by using the mortar pestle. Taken five gram of dried leaf powder was added 100 ml of distilled water and kept for boiling at 80^0C for 30 minutes. After the extract was cooled at room temperature, then filtered by (Whatman No. 1 filter paper) the filtrate was stored for further experimental analysis.

2.3 Preparation of Nanoparticles

A known amount (20 ml) filtered leaf extract of 0.1 concentration Zn $(NO_3)_2.6H_2O$ was dissolved in 100 ml of distilled water and heated 60^0C temperature in controlled hot plate in stirring. Then 20 ml of water hyacinth plant leaves extract was added drop by drop with the aid for the color changed. After continues stirring for 3hrs. A light yellow ZnO NPs powder was exposed to annealing in a muffle furnace at 400°C for 2h.

For the synthesis of ZnO nanoparticles, 0.1 concentration both of zinc nitrate hexahydrate $[Zn(NO_3)_2.6H_2O]$ and $[Cd(NO_3)_2 .4H_2O]$ cadmium nitrate tetra hydrate was prepared in 100 ml of distilled water and 20 ml of plant aqueous extract was added drop by drop with the aid for brown color changed taken and kept on magnetic stirrer 60^0 to 80^0 C for 3h duration the NPs were obtained by in muffle furnace at 500^0 C for 2 h. The calcinated powder light black color nanoparticles were the used for further study. The resulting nanoparticles were used for further characterizations and applications.

2.4 Characterization studies of ZnO NPs and CdO-ZnO Nanocomposites

UV absorption spectrum was used to characterize synthesized ZnO nanoparticles. After being sonicated for uniform dispersion, the sample was examined for optical band gap (Eg) using a UV-Vis spectrophotometer at room temperature (UV-1800,Shimadzu). The wavelength of the spectra was between 200 and 800 nm. The chemical composition was studied by using FTIR spectrometer (Thermo Nicolet 380). The FTIR spectra of ZnO doped CdO-ZnO NPs were taken to the IR sample cell between the range of 4000 cm^{-1} and 400 cm^{-1}. The shape and structures of the products were characterized by using scanning electron microscopy (Model JSM 6390LV, JOEL, USA). The surface morphology of the ZnO NPs and CdO-ZnO and the coated cotton fabric was investigated through the scanning electron microscope (SEM). The elements bound on the fabricated ZnO and CdO-ZnO coated cotton fabric was investigated by energy dispersive X-ray (EDX), which is equipped with the SEM.XRD was used for the phase structure and characterization of crystalline ZnO NPs and CdO-ZnO nanocomposite using Cu Ka radiation with 2theta ranging 20 -80^0. Phase purity and grain size were determined by X-ray diffraction (XRD) analysis recorded by diffractometer (PANalytical x'pert PRO Powder X-ray Diffractometer).

2.5 Chemical composition

One of the most serious diseases in the world and one of the biotic factors impacting the productivity of cassava (*Manihot esculenta Crantz*) is root rot. On the other hand, microorganism's that are helpful to plants, such as bacterial entophytes that promote plant growth, have gradually replaced chemical pesticides and industrial fertilizers, which have a significant harmful effect on the environment. In order to determine whether entophytic bacteria associated with cassava roots have the potential to inhibit the growth of *Phytopythium sp.* and stimulate plant growth, our primary goal in this study was to extract and describe these bacteria. A total of 21 endophytic bacteria were found to have different solubilization capacities for the following fertilizers: T_4 - water control; T_3 - chemical fertilizer; T_2 - CdO-ZnO nanocomposites and T_1- ZnO NPs.

2.6 Nano fertilizer

The biomass of cassava plants that were inoculated with T_3 - chemical fertilizer isolate and T_2 – CdO-ZnO isolates, T_4 –Water control, and T_1 -ZnO NPs by foliar spray and substrate irrigation increased. Additionally, T_3 - chemical fertilizer with T_2 –CdO-ZnO nanocomposite isolates exhibited higher biomass. Thus, as they showed cassava endophytic bacteria capable of promoting plant growth as well as suppressing growth of the pathogen that causes soft root rot of cassava in the Tamil Nadu, India region, our findings may help to promote sustainable agriculture.

3 Results and Discussion

3.1. UV-Visible and Fourier transforms infrared spectroscopy (FTIR) of ZnO NPs and CdO-ZnO synthesized using water hyacinth (*Eichhornia crassipes*)

ZnO and CdO-ZnO nanofertilizer made by the leaves of water hyacinths have UV-visible spectra. In fact, the ZnO produced by nanoparticles has a band gap that is 4.09 eV in utilizing energy. These values were acquired by extrapolating the linear component of the curve $(\alpha h\grave{\upsilon})^2$versus $(h\grave{\upsilon})$ to the X-axis using the Taucplot, which gives the band gap energy directly as shown in Figure 1 (a). In this graph, a represents the absorption coefficient and ht represents the photon energy [15]. The characterized by a wavelength of green synthesized ZnO and Cd/ZnO nanoparticles. The graph of the UV Vis spectrum of the produced ZnO nanoparticles shows the absorption maximum peak occurred at 270 nm. *Itimpliesas pectralshiftin* favour of the blue line [16]. ZnO and Cd/ZnO nanoparticles are present in the absorption band that is slightly blue-shifted to 280 and 320 nm.

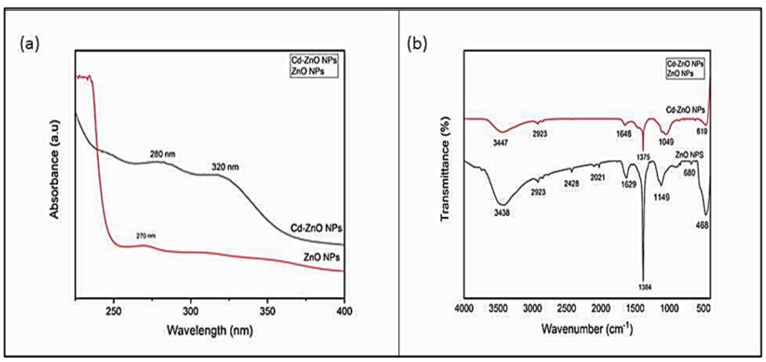

Fig 1. (a) UV-Vis Spectra of ZnO and CdO-ZnO synthesized using water hyacinth (*E. crassipes*); (b) FTIR Spectrum of ZnO and CdO-ZnO NPs synthesized using water hyacinth (*E. crassipes*)

Figure 1 (b) depicts the different FTIR spectra of ZnO and CdO-ZnO nanoparticles. This method was utilized to prepare zinc oxide nanoparticles and cadmium oxide nanoparticles as well as characterize the irrational bands of the water hyacinth leaf extract. A represents the FT-IR spectrum of the zinc oxide and cadmium-zinc oxide nanoparticles had Water hyacinth leaf extract in which the hydroxyl groups of O-H stretching mode are inferred from the broad absorption peak seen at 3438-3447cm^{-1}. The absorption peaks were seen from the range at 2923 cm^{-1}, 2428 cm^{-1}, 2021 cm^{-1} presence of C-H stretch, C-O-C stretch due the presence alkenes group, carboxyl group, ether group. The absorption peak are located it around 1629-1648cm^{-1} representing C=O stretch vibration, C=C stretching vibration. The primary amines [17] vibrations of aromatic groups are responsible for the vibration bands observed at 1384-1375cm^{-1}. The 1149-1049 cm^{-1}band shows the presence of C-O stretching in alcohol and other group compounds. A sharp intense spectroscopic band located around 468cm^{-1} was attributed to the characteristic Zn-O vibrational mode, which confirmed the structure and purity of the synthesized NPs respectively [18]. The ZnO NPs stretching vibration is confirmed by the assignment at 680 cm-1, which corresponds to the C-N stretching vibration, and the absorption peak at 468 cm^{-1} [19]. While CdO-ZnO stretching has a characteristic absorption peak at 619 cm^{-1} [20]. Finally, the vibration bands that were seen in the low-frequency ZnO areas revealed that doped CdO-ZnO nanocomposites had formed

3.2 Morphological characterization of ZnO and CdO-ZnO synthesized using water hyacinth SEM with EDX analysis

Figure 2 Surface morphology of produced ZnO and CdO-ZnO NPs is studied using a scanning electron microscope (SEM), which clearly demonstrates that the particles are nearly cubic and hexagonal spherical in shape, with clear separation and very less agglomeration. Similar findings from biosynthesized ZnO NPs have been reported. An energy dispersive X-ray spectrum (EDAX) from one of the densely populated areas of ZnO NPs was captured in the spot profile mode. With significant signals from the atoms of zinc and oxygen, as well as a very weak signal from the carbon atom, the EDX spectrum of produced ZnO NPs reveals their chemical composition and verifies their purity[21]. The findings demonstrate normal distribution of ZnO and Cd/ZnO NPs doping as well as dispersion of nanoparticles with reduced agglomeration. The preparation of the green synthesis method is confirmed by the presence of Zn+, Cd+, and O- in EDX spectra.

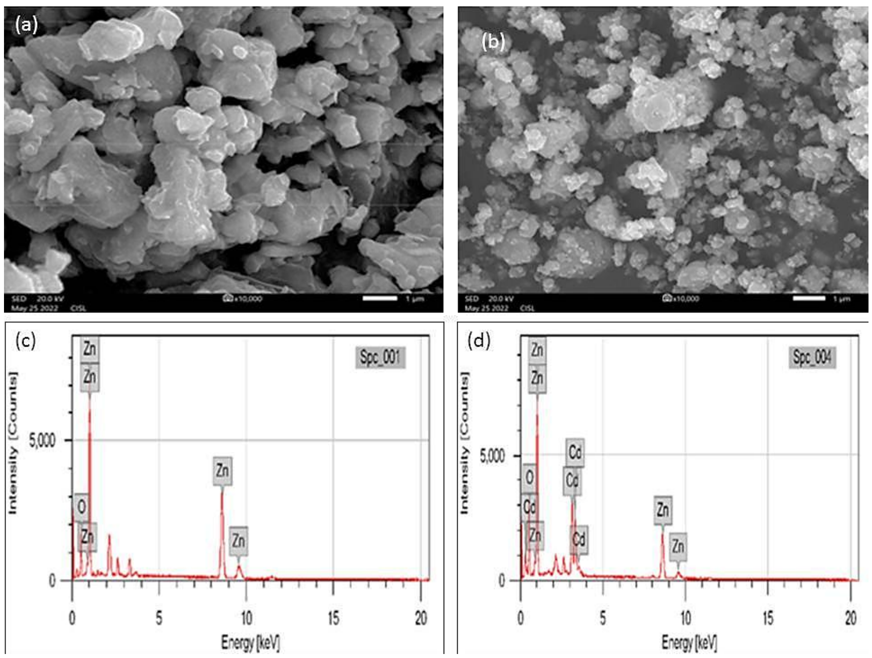

Fig 2. (a, c) SEM with EDX Spectrum images of ZnO doped NPs Synthesized using Water hyacinth leaf extract. (b, d) SEM with EDX Spectrum images of doped Cd/ZnO NPs Synthesized using Water hyacinth leaf extract

3.3 X-ray diffraction (XRD) analysis of ZnO and CdO-ZnO nanocomposite synthesized using water hyacinth

Figure 3 depicts the XRD pattern of ZnO and Cd/ZnO NPs, which revealed a hexagonal wurtzite and cubic structure in accordance with JCPDS card no (89-7102) bimetallic JCPDS Card No (05-0640).The peaks at 31.69^0, 34.35^0, 36.17^0, 47.48^0, 56.48^0, 62.76^0,66.21^0, 67.83^{0}and 31.41^0,34.13^0,47.25^0, 56.26^0, 62.61^{0}correspond to ZnO and CdO-ZnO nanoparticles at 2 theta position . The produced ZnO NPs maintained their hexagonal wurtzite structure and cubic structure nanocomposite with cadmium-zinc oxide nanoparticles, as seen by the intense and hard peaks in the X-ray diffractometer. The small broadness in major peak increases with the addition doping. Using the Debye-Scherrer Equation [22], the crystallite size of ZnO and Cd/ZnO nanoparticles was determined from the XRD data.

$D= K\lambda / (\beta \cos\theta)$,

Where λ is the wavelength of Cu Kα radiation (1.5406 A^0), β is full width half maximum (FWHM) of (101) plane and θ is Bragg's diffraction angle. The average particle sizes of ZnO and CdO-ZnO nanoparticles came out to be 36 nm and 24 nm respectively.

Similarly, Robina Ashraf[23] synthesized undoped ZnO and Fe-ZnO nanoparticles in which the particles size decreased from 29 nm to 19 nm with increase in concentration of iron content. Also, Falak Naz[24] synthesized undoped and Chromium doped ZnO NPs in which the XRD size of nanoparticles decreased with chromium doping from 30.07 nm to 28.14 nm respectively

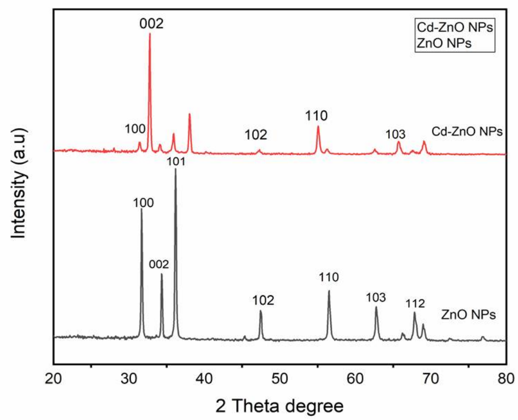

Fig 3. XRD patterns of ZnO and CdO-ZnO synthesized using water hyacinth (*E. crassipes*)

However Hameed[25]. Used silver as doping materials at which with doping the particles size increase from 31.2 nm to 35.3 nm respectively.

3.4 Chemical composition of ZnO and CdO-ZnO nanocomposites synthesized using water hyacinth (*E. crassipes*) derived by the cassava plant

Figure 4 shows the kallakurichi (Dt) chinnaselam (Tk) Kachirapalayam site, Tapioca (*Maravalli kizhangu*) cassava, Kingdom: Plantae, Family: *Euphorbiaceae*, Species: *Manihotesculenta*. Normally, there are two types of Cassava varieties bitter and sweet cassava varieties. Cassava is referred to as "sweet" it is less poisonous. Cassava contains large quantities of cyanide compounds, which must be processed out of the tubers before they can be safely eaten. Application of different nanoparticles using to improved yield components of cassava and at 66% of the current recommended ZnO and CdO-ZnO nanocomposite rate for cassava in the region in the presence of fertilizers. Cassava grown in plots of 5 m$\times$10 m at a planting distance of 30cm$\times$50 cm was hand-harvested according to respective treatments, starting 105 days after planting. Foliage from the control treatment(285 days) and all tubers were only harvested at the final harvest 285 days after planting[26]. The yield increase offered by nanoparticles at chemical fertilizer with the same level of zinc oxide and zinc oxide doping cadmium oxide fertilization was up to 12% yield increase, which represents a significant yields increase for the farmer[27]. Stem lengths of 8cm, 12cm, 16cm and 24cm for the varieties 'NASE19' and 'NAROCASS 1' were cut using rotary cutter.

3.5 Nano fertilizer of ZnO and CdO-ZnO nanocomposites synthesized using water hyacinth (*E. crassipes*)

The zinc oxide and cadmium –zinc oxide nanoparticles made from water hyacinth leaf broth are anticipated to have more extensive applications in biomedical[28–31] fields and in the cosmetic industries because the water hyacinth plant extract is environmentally friendly and extremely cost effective. The method that is being presented can be an economical and effective alternative for the large scale synthesis of zinc oxide nanoparticles. The results of the current study showed that some bio fertilizers, such as mixed algae, mixed algae extract, and mixed bacteria species, had an impact on the vegetative growth, yield parameters, chemical composition, minerals, polyphenols, and vitamin C content of cassava tubers when compared to the control group. Nanostructure catalysts will soon be available for usage, increasing the efficiency of many types of fertilizer application, whether it is used to add soil or spray crops, and enabling the use of lower doses.

Cassava is traditionally harvested after a year on the field and in pieces. Both organic and inorganic fertilizer treatments resulted in statistically comparable cassava yields. Treatment -1 average production from inorganic fertilizer[32] was 23.91 t/ ha^{-1}, comparable to treatments -2 yield of 28.91 t/ ha^{-1} and treatment-3 yield of 18. 67 t/ ha^{-1} treatments – 4 yield of 14.58 t/ ha^{-1} from a combination of inorganic and organic fertilizers. From the unfertilized plot, the lowest yield of 7.91 t ha^{-1} was obtained.

Fig 4. Apply from the fertilizers (a) T_1- Water Control, (b) T_2- Chemical fertilizer,(c)T_3 - ZnO NPs, (d) T_4- Cd/ZnO NPs used of cassava plant

Table 1. Effect of difference between chemical fertilizer on ZnO NPs and Cd-ZnO NPs of cassava plant height (cm) , two branches number of leaves and tuber yield (t/ha^{-1})

S. No.	Treatment	Plant Biometrics	Days Interval									
			0	30	60	90	120	150	180	210	240	270
1.	**T_1- ZnO NPs**	Plant Height (cm)	10.50	15.88	30.66	60.72	90.66	120.32	150.58	170.69	190.94	230.77^b
		Number of leaves	-	5.78	11.23	16.27	20.89	27.34	34.78	39.37	45.29	56.37
		Yield (t/ha)	-	-	-	-	-	-	-	-	-	23.91
2.	**T_2 – Cd-ZnO NPs**	Plant Height (cm)	10.50	25.63	50.34	72.43	97.91	134.11	160.27	178.56	199.44	240.86^a
		Number of leaves	-	8.76	14.97	21.34	36.42	48.74	59.37	64.74	76.24	92.58
		Yield (t/ha)	-	-	-	-	-	-	-	-	-	28.91
3.	**T_3 – Chemical Fertilizer**	Plant Height (cm)	10.50	12.37	25.61	50.74	73.37	95.49	120.29	150.13	185.65	200.98^c
		Number of leaves	-	3.47	7.46	11.63	18.24	26.39	35.78	42.18	47.09	50.74
		Yield (t/ha)	-	-	-	-	-	-	-	-	-	18.67
4.	**T_4- Control**	Plant Height (cm)	10.50	11.02	18.69	36.57	68.30	83.15	111.64	135.85	170.73	190.49^d
		Number of leaves	-	-	2.63	3.75	5.47	10.76	16.57	24.79	29.08	33.27
		Yield (t/ha)	-	-	-	-	-	-	-	-	-	14.58

* Mean of three publications
* In a column, means followed by a common letter are not significantly differ at 5% level by Duncan's multiple range test (DMRT)

3.6 Statistical analysis

Statistical evaluation was done using analysis of variance (ANOVA) followed by Duncan's Multiple Range Test (DMRT). The statistical significance was expressed at $p < 0.05$.For analyzing the result of the medicinal studies, the results obtained are expressed as means ± S. D of six rates in each group.

4 Conclusion

For the first time, we have described a facile, inexpensive biological approach for synthesizing zinc oxide and cadmium-zinc oxide nanoparticles using water hyacinth leaf extract in an aqueous solution. Using SEM with EDX and XRD investigation, the samples' structural characteristics are determined. The synthesized samples average particle size ranges between 36 and 24 nm for UV-visible absorption. The vibrational modes of FTIR such as the bending and stretching properties have also confirmed the formation of ZnO and CdO-ZnO nanoparticles. There are many benefits of using green chemistry to create zinc oxide nanoparticles. Nanofertilizers can help solve the problem of feeding an increasing global population at a time when agriculture is facing various environmental stresses. It is important to realize the useful aspects of nanofertilizers and implement its application to modern agricultural practices. The findings open up a wide range of potential applications for using nanoparticles in general plant investigation and agronomy. By enhancing viability, security, and eventually lowering social insurance costs, nanotechnology improves their efficiency and sufficient. The results of the current study suggest that biological fertilization may be used instead of nanofertilization to attain greater safety.

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