



## ECLIPTA ERECTA ASSISTED GREEN SYNTHESIS OF MN DOPED ZINC OXIDE NANOPARTICLES FOR HEAVY METAL ADSORPTION AND ANTIMICROBIAL ACTIVITY

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### Abstract

Nano metaloxides have been considered as an important reactive adsorbent in waste water treatment due to their unique physicochemical properties. This work reports the preparation of eco-friendly nanoadsorbent ZnO and Mn doped ZnO using *Eclipta erecta* plant extract. *Eclipta erecta* effectively reduced the precursor of metal oxide nanoparticles and acts as a surfactant to minimize agglomeration of nanoparticles during synthesis. The prepared samples were characterized by XRD, FTIR, UV-Vis, fluorescence spectra, FTIR, and FESEM analytical techniques. XRD results revealed the hexagonal phase of ZnO and Mn doped ZnO nanoparticles. FESEM analysis revealed that ZnO and Mn doped ZnO are roughly spherical and particles size were in ranging from 80 – 150nm. Heavy metal adsorption efficiency of Mn doped ZnO nanoparticles were analyzed using ICP-OES and compared with undoped ZnO nanoparticles. Results indicate that Mn doped ZnO nanoparticles exhibited an maximum adsorption of heavy metal ions Hg(89.01%), Cr(91.21%), Cd(96.02%), Ni(97.03%) and Pb(97.05%). Experimental results indicate that prepared Mn doped ZnO nanoparticles could be an effective nanoadsorbent for heavy metal removal from waste water.

**Keywords:** ZnO, Mn:ZnO, Nanoparticles, Green synthesis, *Eclipta erecta*, Heavy metal adsorption, antimicrobial activity

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## 1. Introduction

Fresh water is an important resource for human and other living organism in the earth[1]. However, modern industrialization and urbanization increased the pollution in fresh water bodies. Anthropogenic activities and industrial discharge contaminate the fresh water bodies such as lakes, ponds and river which reduces the amount of freshwater that is available[2], [3]. More than 80% of the wastewater generated by diverse human activities worldwide, according to a research by the United Nations Education, Scientific, and Cultural Organization (UNESCO), is released into the environment without receiving proper treatment. The increase in heavy metal contamination in water resources is a result of the use of a wide range of metals in more sectors and the habit of dumping untreated waste into water bodies. Heavy metals, are extremely toxic when it is exposed to the living organism even at low concentration[4]. The exposure of heavy metals to the humans caused serious health issues such as mutagenesis, carcinogenesis, neurological disorders, and multiorgan failure. Sometimes, indirect exposure of heavy metals polluted water also damage ecosystem because of their tendency to accumulate physiologically, which leads to their biomagnification in food chains, there are instances when even a small amount of indirect exposure to HM-polluted water sources can cause significant harm to the ecosystem[5]. Headaches, nausea, anorexia, hepatitis, constipation, sleeplessness, anaemia, kidney damage, miscarriages, encephalopathy, cancer, nephritic syndrome, and other health issues are brought on by heavy metals[6], [7]. Indeed the heavy metal contamination in fresh water is become serious concern. So, mitigation is urgently needed. Research on heavy metal detection and mitigation has been progressing well. At the same time advanced ecofriendly heavy metal removal technique is highly appreciated. Significance research on heavy metal removal is well understood. Various techniques such as membrane filtration[8], [9], flotation[10], ion-exchange process[8], reverse osmosis, oxidation, electrochemical, coagulation, zeolite process etc. have been developed for waste water treatment. Among them adsorption is considered as an effective method for heavy metal removal due to their simple design, high surface to volume ratio, number of reactive sites, ecofriendly nature, efficiency to remove trace level heavy metal ions. In the adsorption process, adsorbent is added directly to the contaminated water where the

adsorbate attached to adsorbent surface sites. As of today's adsorption technique is said to be energy efficient, eco-friendly approach for heavy metal removal from waste water. Metal oxides such as MgO, TiO<sub>2</sub>, ZnO, MnO<sub>2</sub>, Al<sub>2</sub>O<sub>3</sub>, Fe<sub>2</sub>O<sub>3</sub>, CeO<sub>2</sub> are found to be good adsorbent in waste water treatment. Some studies suggest that ZnO based nanocomposites acts as effective nanoadsorbent when compared to other oxides[11]–[13]. ZnO based metal oxide nanocomposites effectively removes heavy metal ions Cd, Hg, Cr, Ni, and Pb in waste water.

ZnO is n-type semiconducting metal oxide and it has been researched widely for various applications such as sensor, antibacterial, energy storage and wound healing[14]. ZnO possesses three different structures viz. rock salt, hexagonal wurtzite, Zinc blende, among them hexagonal wurtzite structure found to be thermodynamically stable at ambient condition. Zn and O share sp<sup>3</sup> hybridized orbital. The electronegativity of Zn and O are 3.44 and 1.65 respectively[15]. Morphology, size of the materials influences the physicochemical properties. So, it is essential to prepare nanoparticles with controlled morphology and size to be efficient for specific application.

ZnO nanoparticles synthesized by hydrothermal method effectively removed Cr ions in dental waste water. Heavy metal Cr ion removal by ZnO is attributed to its effective surface area 26.7m<sup>2</sup>/g as identified from BET studies [16]. ZnO has been synthesized by simple solution route which effectively removed Cu ions from aqueous solution[17]. ZnO often loaded with electrospun polyacrylonitrile nanofiber for removal of Cd and Pb heavy metal in aqueous solution. The composite nanofiber showed best heavy metal adsorption capacity reached equilibrium at 60min and explained pseudo-second-order kinetic model[18]. Incorporation of dopant such as Al ions on ZnO influences the structural and morphological properties of ZnO which eventually enhances the adsorption of Cr, Cd, Ni and Co ions in aqueous solution[19]. Contamination of water with heavy metals and radionuclides has been increased due to anthropogenic and industrial activities. Kovo et al reviewed ZnO nanoparticles and its effective removal of heavy metal ions[20]. Sheela and coworker studied adsorption properties of ZnO and found to be 357, 387 and 714 mg/g for Zn(II), Cd(II) and Hg(II) ions respectively[21]. Humic acid modified ZnO nanoparticles has been prepared and applied for heavy metal removal. Study reported that ZnO had adsorbed Cd<sup>2+</sup>, Cu<sup>2+</sup>, and Ni<sup>2+</sup> effectively. Experimental

data were correlated with Langmuir adsorption isotherm. Graphene modified ZnO was prepared by facile microwave assisted hydrothermal method. The composite had excellent adsorption characteristics of Ni ions in aqueous solution[22]. Biochar has been incorporated into ZnO to increased heavy adsorption characteristics. Biochar has been prepared from cassava root husk and incorporated with ZnO for heavy metal removal in aqueous solution. Results showed that composite had excellent in removal of As(III), Cd(II), Pb(II) and Cr(VI) ions from aqueous solution. Even though the ZnO metal oxide has been successfully applied for heavy metal adsorption, it required lot of

chemicals such as reducing agent, stabilizing agent required during synthesis. The by-products during nanoparticles synthesis affects environment and create harmful effects in their vicinity. So, green approach is required to prepare nanoadsorbents. Plant extract on the other hand offer wide scope for nanoparticles synthesis. Plant extracts have biomolecules which acts as stabilizing and reducing agent. In such case synthetic chemicals, reducing agent, surfactant might not be required during nanoparticles synthesis. Green chemistry principle is to reduce the use of chemicals for novel compound synthesis



**Figure 1: Eclipta erecta**

Plant extracts has been used for ZnO preparation due to their biomolecules responsible for metal nanoparticles formation. Green chemistry where in plant extract has been preferred for nanoparticles synthesis as this method is ecofriendly, possible large scale production, low cost and prevent from nanoparticles agglomeration[23]–[25]. Eclipta erecta (Figure 1) is an important medicinal herb with lot of medicinal value. This plant extract is been underutilized especially, metal oxide nanoparticles synthesis. In the present work, Eclipta erecta plant has been chosen to prepare ZnO nanoparticles for effective removal of heavy metals.

## 2. Materials and methods

### Materials

Zinc nitrate ( $\text{Zn}(\text{NO}_3)_2 \cdot 6\text{H}_2\text{O}$ ), Manganese

nitrate hexahydrate ( $\text{Mn}(\text{NO}_3)_2 \cdot 6\text{H}_2\text{O}$ ) were procured from sigma Aldrich. Eclipta erecta plants were collected from local market. Millipore water ( $18 \text{ M}\Omega \cdot \text{cm}$  at ambient condition) was used throughout the experiment. All necessary chemicals and solvents used in this experiment were analytical and used as received.

### Preparation of plant (Eclipta erecta) extracts

Eclipta erecta plant was thoroughly washed with distilled water and cut into 5-10mm small piece. About 50g of plant was ground using mortar and pestle and transferred into 250ml beaker. Millipore water was added to the ground plant paste with the ratio of 1:10 (plant paste: water) ratio. The mixture was heated at  $80^\circ\text{C}$  for 20 minutes with constant stirring in a magnetic stirrer. The solution was filtered using whatman No.1 filter paper and the filtrate was stored in

clean glass vial and kept at room temperature for synthesis of metal oxide nanoparticles.

Preparation of Mn:ZnO nanoparticles using *Eclipta erecta* extracts

Zinc nitrate (0.1 M) and manganese nitrate (0.01M) were prepared using Millipore water and taken in a 250ml glass beaker. To this, 50ml of freshly prepared *Eclipta erecta* extracts was added to the metal precursor solution. The reaction mixture was heated at 80°C with constant stirring for 3 hours. Clear solution to whitish brown precipitate formation was observed after 3 hour stirring. The obtained brown precipitate was separated using centrifuge with 16000rpm and dried at 120°C for 6 hours. Then the powder sample was stored in a glass vial for further application. Similar protocol was followed for ZnO nanoparticles preparation without addition of dopant Manganese nitrate hexahydrate.

Characterization

X-ray analysis of the prepared samples were done using Model:D-8 Advance Bruker AXS. Hitachi UH5300 spectrophotometer was used to record UV-visible spectrum of ZnO and Mn doped ZnO samples. Shimadzu Fourier Transform Infrared Spectroscopy (FTIR) 8400

Spectrometer was used to confirm the functional

groups of as prepared ZnO NPs. For analysis, the dried powdered sample was mixed with KBr and pressed (11,000 psi) to pellet disc. Nitrogen gas was purged to detector to minimize the error related to moisture and increase the signal level. The pellet was inserted on sample compartment and spectrum was recorded in the range of 4000 to 400  $\text{cm}^{-1}$ . Cary eclipse fluorescence spectrophotometer (Make:Agilent) was used to analyze emission spectra of the samples. Surface morphology and elemental analysis of the sample was observed using Field emission scanning electron microscope (FESEM), Model & make - CARL ZEISS (USA) , MODEL: SIGMA, Resolution 1.5 nm.

Adsorption studies

For heavy metal adsorption studies, batch mode method was conducted, NaOH and HCl were used to adjust pH of the solution. Aqueous solution containing heavy metal ions were mixed with 100mg of prepared adsorbent separately and contact time allowed to 30, 40, 60, 80, 120, 160 minutes respectively. ICP-OES instrument (Make: Thermo Fisher Scientific, Model: iCAP 7400) was used to analyze heavy metal concentration. The heavy metal adsorption per unit mass of Mn:ZnO and ZnO nanoparticles were determined by following equation Eq. (1) and the removal efficiency of the sorbent was calculated using Eq. (2).

$$(1) \quad q_e = \frac{(C_0 - C_a)}{m} \cdot \frac{V}{1000}$$

$$(2) \quad E(\%) = \frac{(C_0 - C_a)}{C_0} \cdot 100$$

Where,  $C_0$  is the stock solutions initial concentration,  $C_a$  is the final concentration of stock sample after adsorption (mg/L),  $q_e$  - Amount of heavy metal adsorbed into the AC (mg/g),

$V$  - Volume of the medium (mL) ,

$m$  - Amount of the AC (g)

$E$  - Removal efficiency (%)

### 3. Results and discussion

Structural properties of ZnO and Mn doped ZnO nanoparticles were performed by X-ray diffraction analysis. The XRD results obtained is presented in figure 1. Figure 1 shows the XRD pattern obtained for ZnO and Mn doped ZnO nanoparticles prepared using *Eclipta erecta*

Extracts. Distinct peaks observed at 31.7°, 34.4°,

36.4°, 47.5°, 56.8°, 62.7°, 66.5°, 68.1°, 69.2°, 72.5°, 77.1° can be indexed to miller indices (100), (002), (101), (102), (110), (103), (200), (112), (201), (004), and (202) respectively. The theta values are matched with standard JCPDS no. 36-1451 and it is clearly indicated that the ZnO and Mn doped ZnO are formed with wurtzite structure, hexagonal phase. Further the crystallite size of the samples was evaluated using Scherrer formula as given in equation (1).

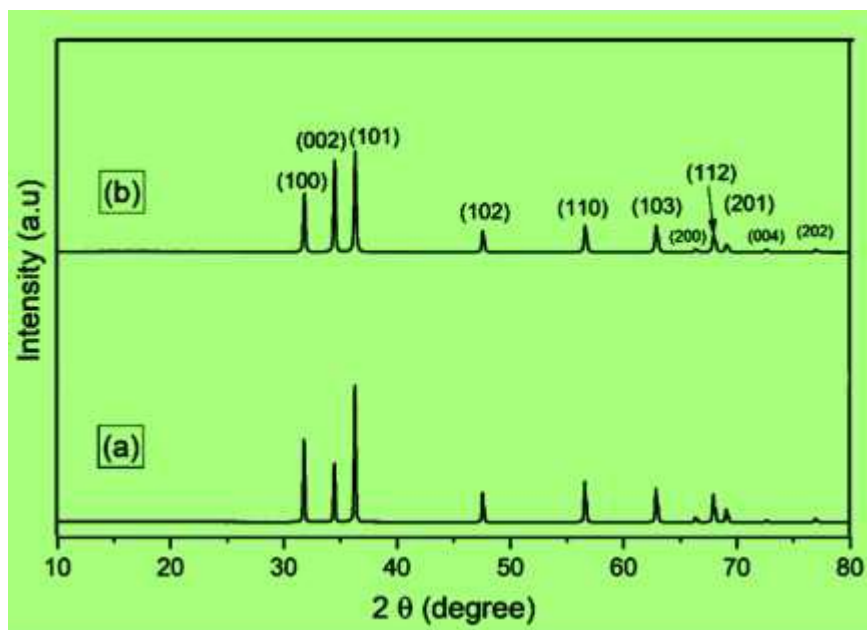


Figure 1 XRD pattern obtained for (a) ZnO and Mn doped ZnO nanoparticles

These distinct clear peaks indicated the crystalline nature of ZnO obtained through plant extracts. Crystallite size ( $D$ ) was calculated using Scherrer's formula as given in equation (1)

$$D = \frac{0.9\lambda}{\beta \cos \theta} \quad (1)$$

Where,  $\beta$ - full width half maxima of a peak,  $\lambda$  – incident X-ray wavelength. Average crystallite size was calculated for the sample ZnO and Mn doped ZnO nanoparticles which are found to be 32.5nm, and 30.2nm respectively. The difference in crystallite size of ZnO and Mn:ZnO are due to plant biomolecules and  $Mn^{2+}$  influences the lattice of host materials.

UV-Visible spectroscopy revealed the optical characteristics of metal oxide nanoparticles. Due to surface plasmon resonance effects, electron present in nanoparticles will start to oscillate at

certain wavelength. Figure shows the UV-Vis spectra of (a) ZnO and (b) Mn doped ZnO nanoparticles synthesized using *eclipta erecta* plant extracts. The lambda max of ZnO and Mn

ZnO showed maximum absorbance at 378nm and 377nm respectively, indicate that synthesized particles are photosensitive in the UV-vis region.

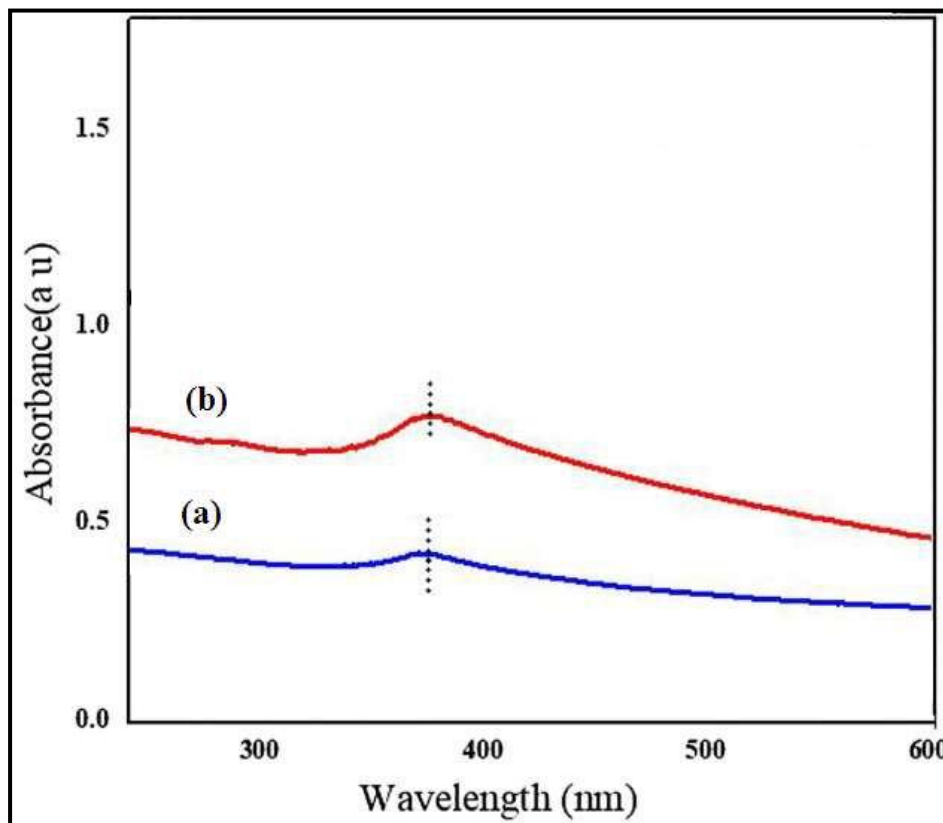


Figure 2 UV-Vis absorbance obtained for (a) ZnO and Mn doped ZnO nanoparticles

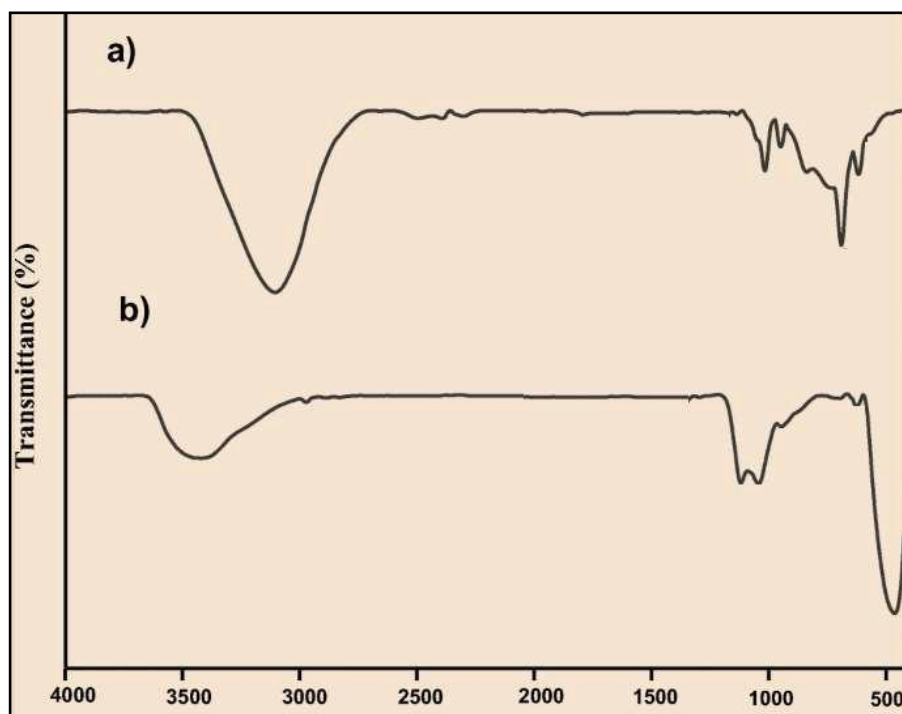


Figure 3: FTIR spectra of (a) ZnO (b) Mn doped ZnO nanoparticles

FTIR spectra were carried out to determine the biomolecules functional groups on metal oxide nanoparticles. The FTIR spectra were acquired in the range of  $4000\text{cm}^{-1}$  to  $400\text{cm}^{-1}$ . The peaks at  $3445\text{ cm}^{-1}$  and  $3102\text{ cm}^{-1}$  indicate that stretching vibration of (OH) hydroxyl group. The peaks observed at  $1041$  and  $1116\text{ cm}^{-1}$  due to C-O stretching vibration. The peaks observed

at  $950$ ,  $946$  and  $693\text{ cm}^{-1}$  due to  $=\text{C}-\text{H}$  bending vibration. The strong peaks observed at  $568$  and  $465\text{ cm}^{-1}$  attributed to strong interaction between Zn and oxygen species formation of ZnO nanoparticles. Results indicate that plant extract has biomolecules which is responsible for metal nanoparticles formation.

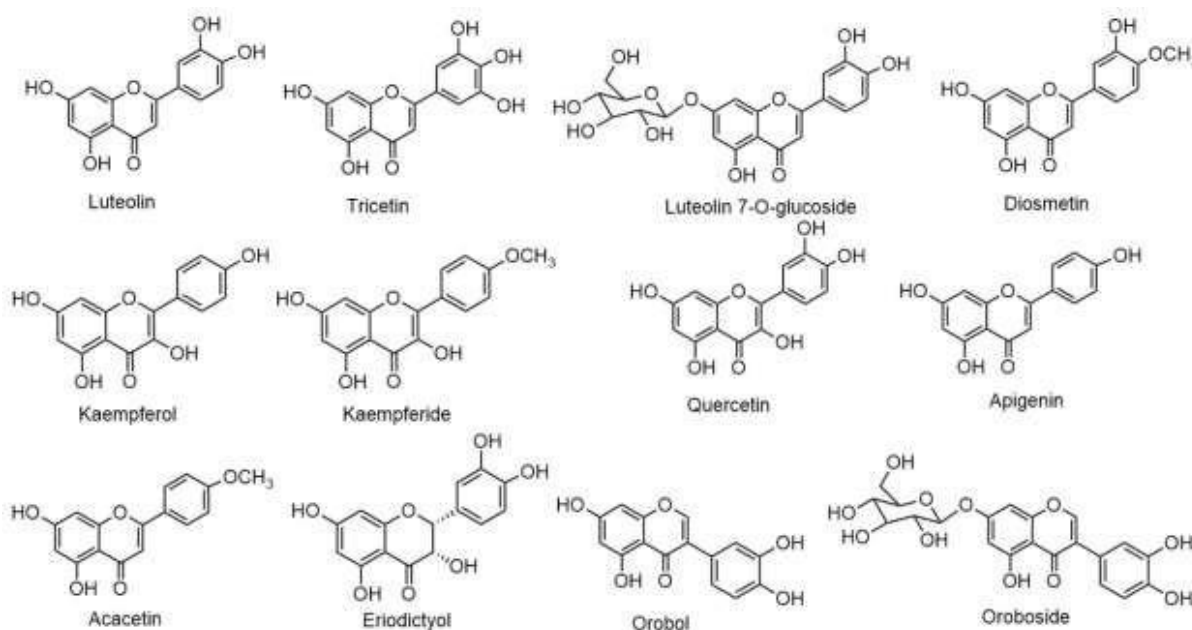


Figure 4: Structure of major flavonoids present in *Eclipta erecta* (Source:[26] )

The plant extract has important flavonoids as shown in figure 4, which may be act as reducing agent during nanoparticles synthesis.

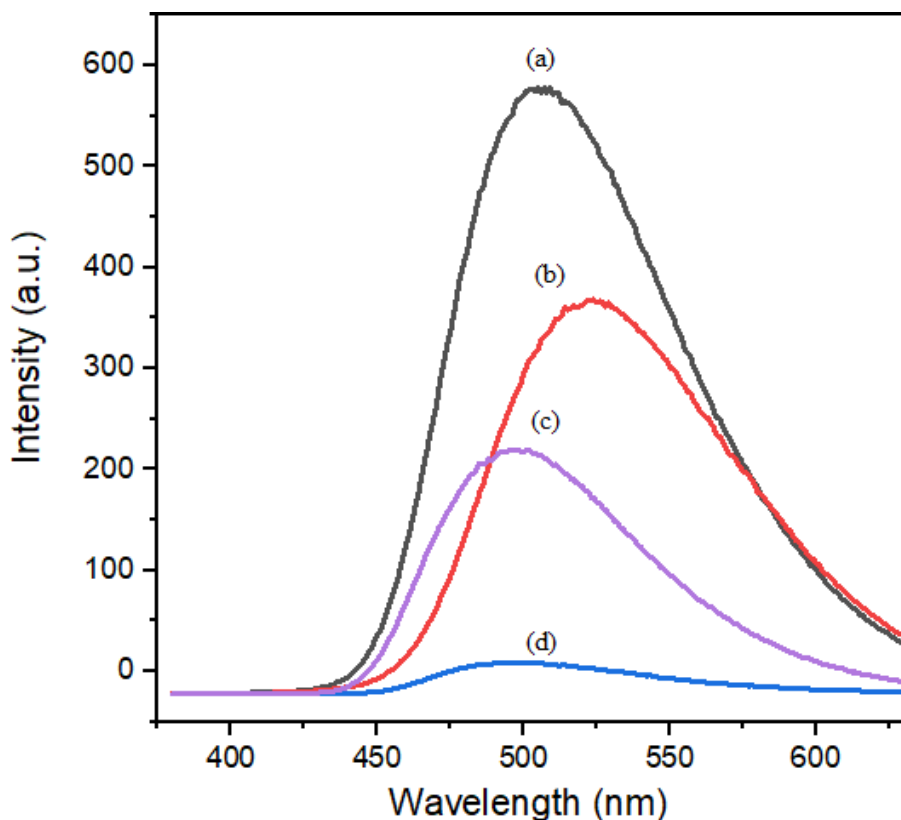


Figure 5: Photoluminescence spectra of (a,c) Mn:ZnO, (b,d) ZnO nanoparticles with different wavelength excitation

Figure 5 shows the photoluminescence (PL) characteristics of ZnO and Mn doped ZnO nanoparticles excited at different wavelength 350nm and 360nm respectively. PL spectra of ZnO (Figure b, d) exhibit emission at 495nm and 497nm. Similarly Mn doped ZnO nanoparticles exhibit emission at 493nm and 496nm. Slightly blue shift observed when the nanoparticles excited at different wavelength. The blue shift is attributed due to quantum confinement effect. Further Mn doping on ZnO host material enhances the surface active sites which could enable more heavy metal ions adsorptive characteristics. Nanoparticles are typically having excellent

physicochemical properties when compared to their bulk materials. For heavy metal adsorption application, the size of nano adsorbent should be within the range of nanoscale and controlled morphology. These features influence the adsorptive characteristics. Figure shows the scanning electron microscope (SEM) image obtained for ZnO and Mn:ZnO synthesized using *Eclipta erecta* plant extract. It is observed that ZnO nanoparticles Figure (a) and Mn:ZnO figure (b) formed roughly spherical and slightly agglomerated. The size of the nanoparticles is found between 80 to 200nm which confirmed the formation of nanoparti



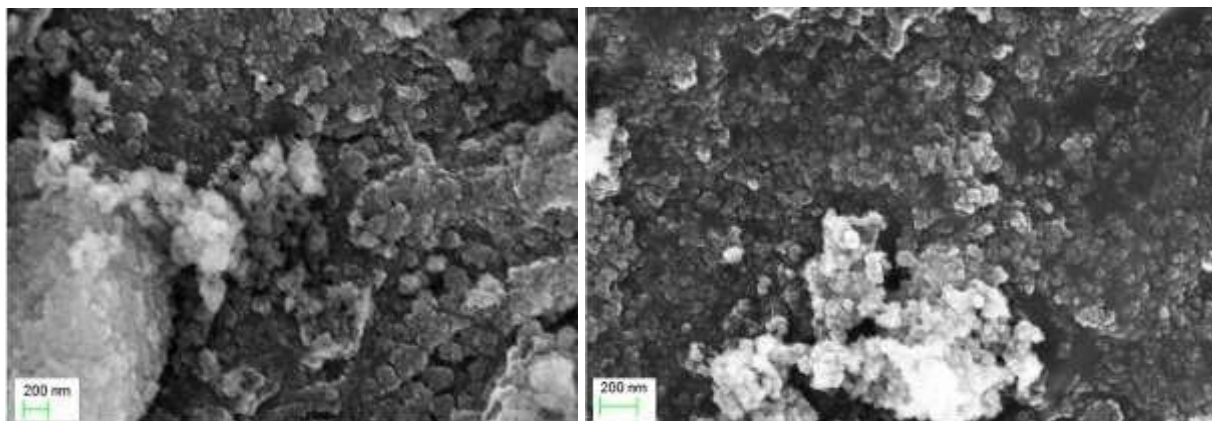


Figure 6: SEM images of (a) ZnO and (b) Mn:ZnO nanoparticle Adsorption studies

Nanoparticles size and morphology is the key factor in fine tuning the adsorption capacity to remove heavy metals from their aqueous aliquots. The present experimental results demonstrates that the Mn doped ZnO nanoparticles prepared using plant extract exhibited better results in comparison with

previous similar research reported[1]. The SEM analysis revealed the nano size of the particles which has the high surface area accessible for the adsorbate-adsorbent interaction will increase in proportion to the lower size nanocomposites. In addition, the high surface to volume ratio decreases the path inside the pores of ZnO nanoparticles.

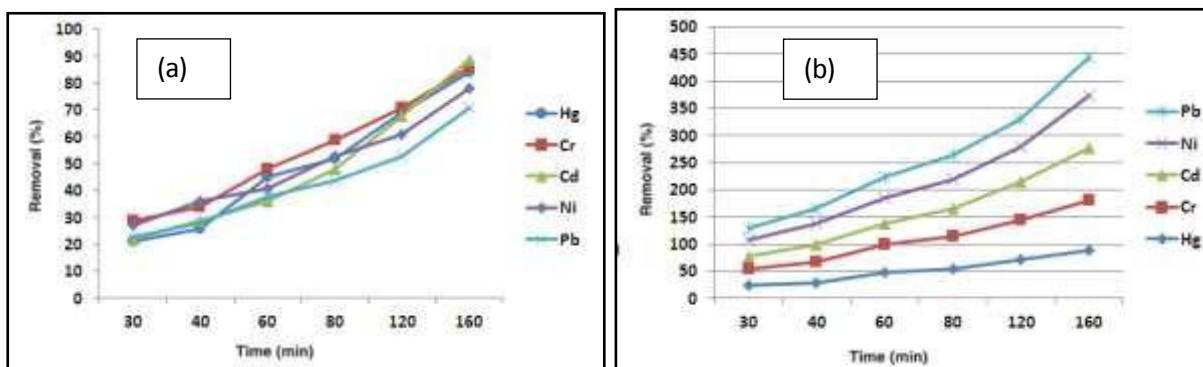


Figure 6: Effect of contact time on adsorption of heavy metal ions on adsorbent (a) ZnO and (b) Mn doped ZnO nanoparticles. (initial conc = 100mg/L, adsorbent dosage: 2 g L<sup>-1</sup> , 100 rpm)

Figure 6 shows the Effect of contact time on adsorption of heavy metal ions on adsorbent (a) ZnO and (b) Mn doped ZnO nanoparticles. It is observed that Mn doped ZnO nanoparticles exhibited an maximum adsorption of heavy metal ions Hg(89.01%), Cr(91.21%), Cd(96.02%), Ni(97.03%) and Pb(97.05%). However, different parameters such as adsorbent/adsorbate concentration, pH and temperature need to be studied to optimize the nanoadsorbent for heavy metal removal.

#### 4. Conclusion

*Eclipta erecta* plant extract has been used to synthesize Mn doped ZnO nanoparticles by facile wet chemical approach. XRD and SEM analysis revealed the nanoparticle nature and FTIR confirmed the biomolecules of plant extract responsible for nanoparticles formation. Photoluminescence spectra revealed the quantum confinement nature of Mn doped ZnO nanoparticles. Mn doped ZnO exhibited maximum heavy metal removal capacity than ZnO nanoparticles. Experimental results indicate that prepared Mn doped ZnO nanoparticles could be an effective nanoadsorbent for heavy metal removal from waste water in the near future. However, different parameters such as adsorbent/adsorbate concentration, pH and temperature need to be studied to optimize the nanoadsorbent for heavy metal removal.

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