



Green Synthesis of ZnO Nano Particles, its Characterization and Application

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Abstract

Nano ZnO was synthesised by green approach employing aqueous extract of Adulsa and Lemongrass leaves. XRD suggested hexagonal wurtzite structure for these prepared ZnO. Synthesised nanoparticles efficiently catalysed Biginelli reaction.



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Introduction

Nanoparticles are gaining importance in various fields such as health, biomedical science, chemicals industries, food & feed, cosmetics, environmental, drug and gene delivery, energy science, electronics mechanics and space industries.¹⁻²

Design of processes that reduces or eliminates generation of hazardous substances is the key principle for sustainable chemistry. Developing green methods for synthesising nanoparticles is a major focus of present research scenario.³⁻⁷

Available literature forecasts that the nanoparticle synthesis using medicinal plants, micro-organisms and algae and other as source has been unexplored and underexploited.⁸⁻¹⁶

Materials and Methods

Preparation of Extract

A fresh leaf of Adulsa or Lemongrass leaves were collected and washed several times with deionized water, grinded and boiled for 30 min. till the colour of solution changes from watery to light yellow and filteres to get the extract. The filtrate is used as a stabilizing (capping) agent.

Experimental Procedure

For the synthesis of nanoparticles, Adhatoda/ Lemongrass leaves extract (50 mL) was taken and heated to 80 °C with vigorous stirring. $\text{ZnNO}_{3.6}\text{H}_2\text{O}$ (5g) was added to the solution under heating. The paste thus obtained was heated in ceramic crucible at 400 °C for 3 h in air. Nano ZnO was obtained as white powder.¹⁷⁻¹⁸

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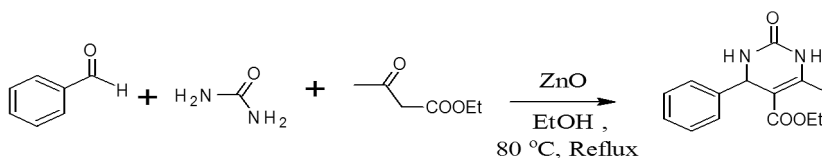
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Characterisation

XRD analysis was performed on Philips XPert Pro diffractometer. JEOL JSM-6360LV Scanning Electron Microscope was used to study surface morphology of nanoparticles. FTIR were recorded on ThermoFischer FTIR instrument with KBr.²¹⁻²²

Catalytic Application

Urea (15 mmol, 0.90 g), Ethylacetoacetate (13 mmol, 1.69 g), Benzaldehyde (10 mmol, 1.06 g), prepared ZnO catalyst (0.2 g), Ethanol (10 mL) were refluxed for 3 hr. (Scheme 1) Mixture was then filtered in crushed ice and crude solid product as separated



Scheme 1: Biginelli Reaction.

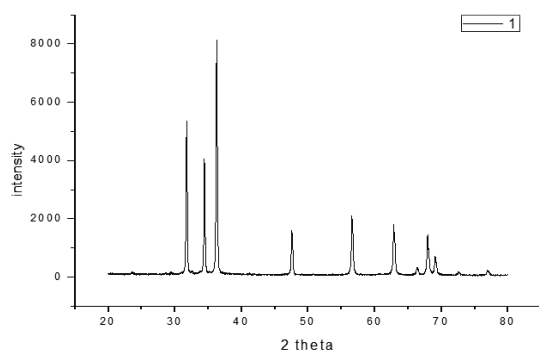


Fig. 1: XRD pattern of ZnO (Adulsa)

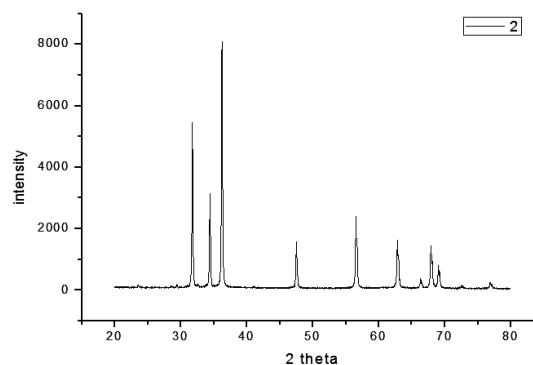


Fig. 2: XRD pattern of ZnO (Lemongrass)

by filtration and washed with water. Product was purified by recrystallisation with Ethylacetate and structure was confirmed by comparing melting point and IR data.²³⁻²⁵

Result and Discussion

X-ray Diffraction (XRD)

The technique provides valuable information of structure, different phases and preferred crystal orientation. The pellets sintered at 400 °C were finely powdered and used for XRD analysis. Inter-atomic spacing (d), lattice parameter (a), and particle size (D) were calculated for the highest intensity peak having miller indices as (hkl) for the above sample.

67.95° (112), 69.01° (201) and 78.82° (202). These peaks correlate to the hexagonal wurtzite structure of ZnO without any impurity peaks. The average crystallite/particle size as calculated by Debye-Scherrer equation was found to be 28 nm.

Fig. 2 shows XRD pattern for ZnO prepared using Lemongrass leaves extract. These peaks are indexed as 31.74° (100), 34.40° (002), 36.23° (101), 47.52° (102), 56.59° (110), 62.82° (103), 66.30° (200), 67.88° (112), 69.00° (201) and 76.93° (202). This pattern also correlates to the Hexagonal wurtzite structure of ZnO. The average crystallite/particle size was found to be 50 nm.

Scanning Electron Microscopy (SEM)

Fig. 3 and 4 shows SEM images depicting the morphologies of nano ZnO obtained by Adulsa and Lemongrass resp.

Figure 3 shows the formation of irregular spherical ZnO nanoparticles, and change of the morphology of the nanoparticles. This SEM image indicates irregular morphology. Some particles are in the range of 85-95 nm.

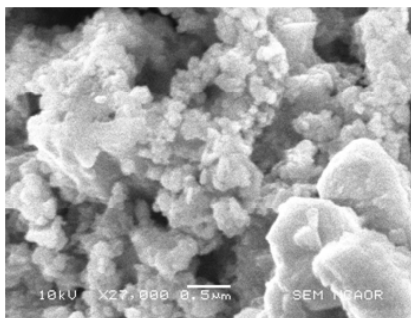


Fig. 3: SEM Images of ZnO (Adulsa)

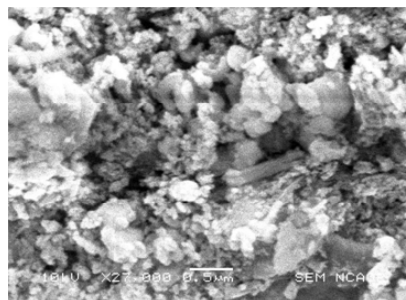


Fig. 4: SEM Image of ZnO (Lemongrass)

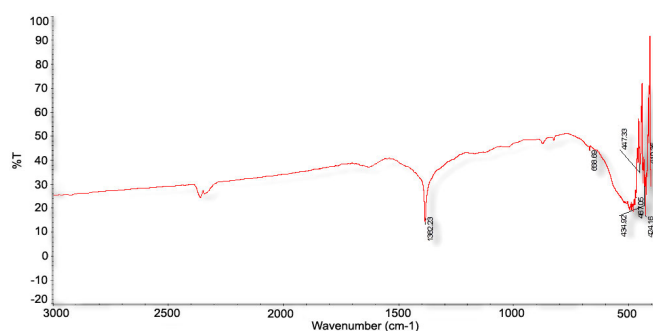


Fig. 5: FTIR spectra of ZnO (Adulsa)

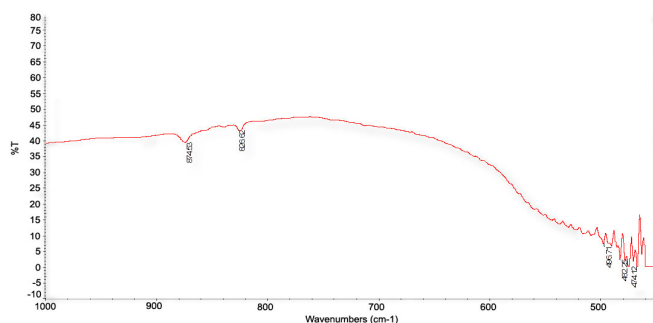


Fig. 6: FTIR spectra of ZnO (Lemongrass)

Figure 4 shows the formation of spherical ZnO nanoparticles, and changes of the small granular size of zinc oxide nanoparticles are formed. Spherical morphology of the nanoparticles, mixed with some chunky particles due to agglomeration. It is seen from the image that the zinc oxide nanoparticles range from 85-98 nm and there are also some nanoparticles are formed in the range of 500nm.

Figure 5 and 6 shows the FTIR spectrum of the ZnO nanoparticles synthesized by green method, this spectrum shows accordance with the literature spectrum. IR graph depict that the adhatoda(adulsa)

extract acts a stabilization(capping agent). Adhatoda sample is rich in alkaloids, tanins, saponins, phenolic, and flavonoids. The chemical constituents of lemongrass extract areterpene associated to aldehyde, alcohol and ketones. All these constituent promote these lemongrass extract as a stabilizing agent. These biomolecules helped in stabilization (capping) of the synthesized ZnO NPs.

Catalytic Activity

Table 1 presents the performance of prepared ZnO catalyst for Biginelli reaction giving dihydropyrimidinone product. (M.P. 207 °C, Lit. 206-208 °C)²³⁻²⁵

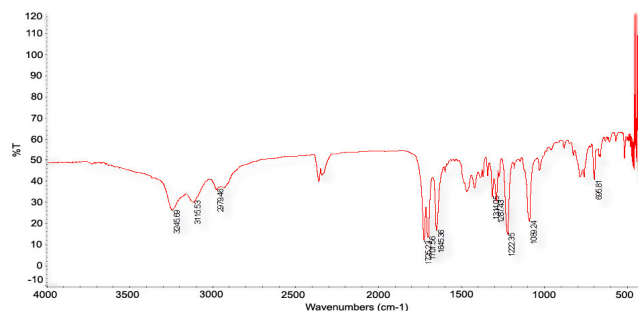


Fig. 7: FTIR spectrum of dihydropyrimidinone

Table 1: Catalytic activity

Catalyst	Time (Min)	% Yield
ZnO (ADULSA)	180	64
ZnO(LEMONGRASS)	180	75
COMMERCIAL ZnO	180	66

Table 2: Characteristics bands.

Observed Peak cm ⁻¹	Stretching mode
3245	N-H asymm
3115	N-H symm
2979	C-H
1725	C=O Ester
1701	C=O Amide
1645	C=C
1089	Monosubstituted Aromatic Ring

Fig. 7 displays the IR spectrum of dihydropyrimidinone product obtained from Biginelli reaction while Table

2 correlates its characteristic absorption peaks with probable vibrational modes.

Conclusion

In this study we report a simple, biological and low cost approach for nano Zinc oxide using various leaves extract asadhatoda(adulsa), lemongrassas the reducing agent. They are employed as stabilizing agents. The prepared Zinc oxide nanoparticles were characterized by XRD, SEM and FTIR. XRD patterns of ZnO nanoparticles prepared with the green synthesis method using leaves extracts of adhatoda(adulsa), lemongrass, neem, meethi, tulsi, shows average particles size calculated as in the range of 25-50 nm by using Scherer formula. SEM image indicate irregular morphology with particles are in the range of 85-100 nm. Also, the synthesized ZnO catalyst was used in Biginelli reaction to prepare 3,4-Dihydropyrimidinones from ethylacetoacetate, benzaldehyde and urea with around 75 % product yield.

Acknowledgement

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