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METAL OXIDE CATALYZED AN EFFICIENT GREEN SYNTHESIS OF NOVEL NITROGEN-CONTAINING HETEROCYCLIC COMPOUNDS

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ABSTRACT

Nano-catalysts are catalysts composed of nanoparticles or nanostructures that enhance the reaction rate without being altered by the process. Nowadays, there is a lot of interest in nano-catalysts in organic synthesis. Due to their high catalytic activity, organic synthesis based on nano-catalysts has benefits in the field of green chemistry. Furthermore, nano-catalyst is easily manufactured, recyclable, and biocompatible. Zinc oxide, a well-known inorganic metal oxide in nano-catalyst, shows great anti-bacterial activity. Current experiment emphasizes on prediction of anti-bacterial activity of zinc oxide nano-catalyst. We have also utilized the synthesized nano-catalyst for the synthesis of 1H-indole and (E)-1,3-diphenylprop-2-en-1-one derivatives. Our findings rationalized the effective synthesis of spherical nano-catalyst with an average size range of 1-200 nm. UV-Vis absorption spectroscopy, X-ray diffraction (XRD), Fourier transform infrared spectroscopy (FTIR), and Scanning Electron Microscope with the Energy Dispersive X-ray studies (EDX) information buttress the result generated via XRD pattern in terms of size and purity. The ZnO nano-catalyst demonstrated an anti-bacterial effect in contrast to Escherichia coli (E. coli) that was dose-dependent, with an estimated IC_{50} value of around 20 µg/mL. The study endorses the potential usage of ZnO nano-catalyst in industries like food, pharmaceutical, agriculture, and cosmetic industries for its anti-bacterial activity.

Keywords: Green Synthesis, Characterization, Zinc Oxide Nano-Catalyst, Anti-Bacterial Activity, Ultrasound-Assisted Synthesis.

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INTRODUCTION

Nowadays, there is a lot of curiosity in nano-catalysts/Nano-particles in organic synthesis. The highly effective catalytic activity of nano-catalysts makes them advantageous in the field of green chemistry when used in chemical synthesis. Furthermore, the nano-catalyst is easily fabricated, recyclable, and biocompatible.[1,2] Nanotechnology may be applied in several sectors in a variety of ways, such as through the use of nanoparticles and nanocomposites. In the past decade, research on the application of nanoparticles in science and medicine has grown significantly.[3] Unlike their bulk form, nanoparticles can target the disease most effectively because of their special advantageous electrical, optical, magnetic, anti-inflammatory, wound-healing, and antimicrobial characteristics.[4] Zinc oxide (ZnO) nanostructures have received a lot of interest because of their special qualities, which include optical transparency, electric conductivity, piezoelectricity, and near-UV emission.[5, 6] They also play significant roles in various scientific fields, including chemical sensors, catalysts etc.[7] Therefore, significant efforts and a variety of approaches, like co-precipitation,[8] hydrothermal,[9] thermal evaporation,[10] chemical vapor method (CVD),[11] pulsed laser deposition (PLD),[12] microemulsion,[13] solvothermal,[14] sono-chemical,[15] and solid-state thermal decomposition,[16] have been reported to be utilized during the synthesis of these metal oxide nanostructures. Among these, co-precipitation is a more cost-effective and unique way for producing stable nano-catalyst/nano-particles, and it is much cleaner and economical.[8] The limited solubility of reactants has been seen to cause the majority of organic reactions to proceed slowly in polar or other solvents; nevertheless, the hydrophobic agglomeration of organic molecules has been shown to lower the

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activation energy, according to the Breslow effect.[17,18,19] There has been a lot of interest in metal-based nano-catalyst applications. With their unique properties, zinc oxide (ZnO) nano-catalyst/nanoparticles have notably carved themselves a significant position. Amphiphilic catalysts or surfactants that effectively provide a hydrophobic reaction environment in water are the two methods utilized to solve this issue with the help of ZnO and other similar nano-catalysts.[20] In the current initiative, we have developed ZnO nano-catalyst as an amphiphilic catalyst to proceed with various reactions. Studies have examined characterization and antibacterial activity and provided evidence for its mechanistic approach.

EXPERIMENTAL

Material and Methods

Preparation of ZnO Nano-catalyst

Zinc chloride (0.60 g, 2 mmol) was dissolved in 5 mL of distilled water at room temperature while being continuously stirred by a magnetic stirrer. Drop by drop, with magnetic stirring, sodium hydroxide aqueous solution (1 M) was added until the pH was between 10 ≈and 12. Following the addition, the mixture was shaken for 1 hr at room temperature, during which time a white suspension developed. After filtering out the white precipitate, it was cleaned three times using ethanol and de-ionized water, and it was allowed to dry overnight at 80 °C in a hot air oven.[21,22]

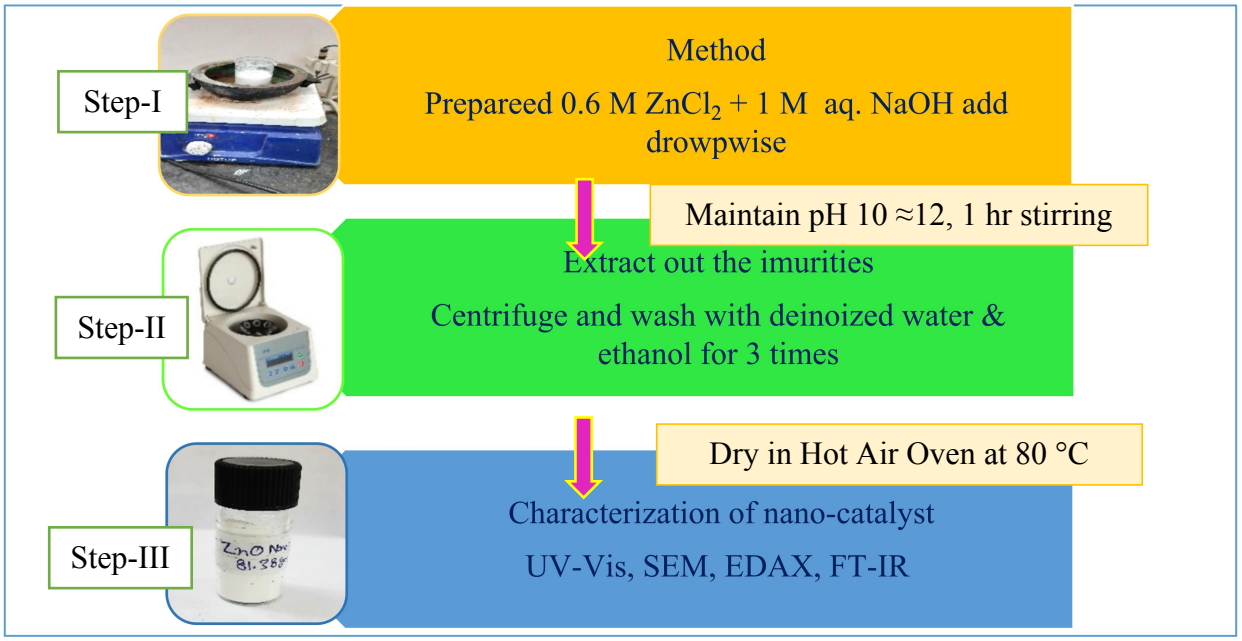


Fig.-1: Schematic Presentation for the Preparation of ZnO Nano-catalyst

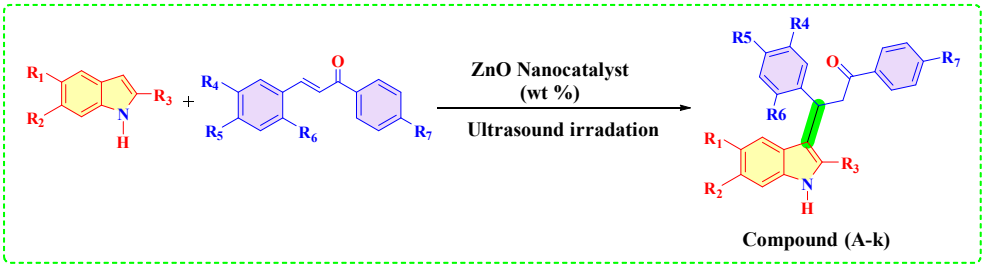
Characterization of ZnO Nano-catalyst

The confirmation of nano-catalyst synthesis has been confirmed by UV-visible measurements after the visible color shift. A scanning electron microscope (SEM) was used to determine the size and shape. Using Fourier Transform Infrared Spectroscopy (FTIR) analysis, the functional groups were found. Using X-ray diffraction analysis (XRD), the crystal structure and lattice plane of the ZnO nano-catalyst were identified. Energy dispersive X-ray analysis (EDAX) was utilized to calculate the elemental structure of the produced nano-catalyst.

General Procedure for the Synthesis of 1H-indole and (E)-1,3-diphenylprop-2-en-1-one Derivatives

Indole (1.293 mmol) and *α, β*-unsaturated ketone or chalcone (1.293 mmol) were weighed and transferred into a 50 ml round-bottom flask. Add 4 ml of acetonitrile as a solvent and zinc oxide nano-catalyst into the reaction mixture. As a green method of reaction facilitation, ultrasonication was employed. The catalyst's mol% and the amount of time needed were then used to fine-tune the reaction conditions (Table-1), considering an equivalent yield. The reaction was monitored by TLC using 20% ethyl acetate: n-hexane.

Using the coupling of indole and chalcone as a model reaction to generate (3-oxoaryl) indole derivatives under ultrasonic irradiation, the catalytic activity of ZnO nano-catalyst was first evaluated (Scheme-1). Our comprehension of zinc oxide nano-catalyst catalytic potential in synthetic chemistry has improved as a result of this scientific method.



Scheme-1: Synthesis of 1H-Indole-Chalcone Derivatives (A-K)

Table-1: Performance of the Reaction Between 1H-Indole-Chalcone Derivatives in the Presence of ZnO Nano-Catalyst

S. No.	ZnO Cat. Mol %	Reaction Time	Solvent used	Yield %
1	2	40 min	Acetonitrile	90
2	4	40 min	Acetonitrile	91
3	6	40 min	Acetonitrile	94
4	8	40 min	Acetonitrile	91
5	10	40 min	Acetonitrile	92
6	No catalyst	60 min	Water	No reaction

Spectral Analysis of Synthesized Derivatives

3-(1H-indol-3-yl)-1,3-diphenylpropan-1-one (A)

Light pink solid. M.P: 161-163 °C. Infrared (IR, 4000-400 cm^{-1}): 3387.53, 2970.32, 1681.19, 1460.86, 1113.97. ^{1}H-NMR (500 MHz, $CDCl_3$) δ: 3.61-3.75 (m, 2H), 4.98 (t, *J* = 7 Hz, 1H), 6.87-7.45 (m, 13H), 7.84 (d, *J* = 7 Hz, 2H), 7.88 (brs, 1H, NH). ^{13}C-NMR (125 MHz, $CDCl_3$) δ: 38.4, 45.4, 111.3, 119.4, 119.5, 119.7, 121.6, 122.3, 126.4, 126.8, 128.0, 128.2, 128.6, 128.7, 133.1, 136.8, 137.2, 144.4, 198.7. Mass (m/Z) ESI method: 324 (M-H). λmax abs 307 nm.

3-(1H-indol-3-yl)-3-(4-methoxyphenyl)-1-phenylpropan-1-one (B)

Light orange solid. M.P: 163-165 °C. Infrared (IR, 4000-400 cm^{-1}): 3396.91, 2989.07, 1648.37, 1451.49, 1390.54, 1292.11, 1118.65. ^{1}H-NMR (500 MHz, $CDCl_3$) δ: 3.58-3.72 (m, 5H), 4.93 (t, *J* = 7 Hz, 1H), 6.69-7.46 (m, 12H), 7.84 (d, *J* = 7 Hz, 2H), 7.88 (brs, 1H, NH). ^{13}C-NMR (125 MHz, $CDCl_3$) δ: 37.6, 45.5, 55.3, 111.2, 114.0, 119.5, 119.7, 119.8, 121.4, 122.3, 126.8, 128.2, 128.7, 128.9, 133.1, 136.5, 136.8, 137.3, 158.1, 198.9. Mass (m/Z) ESI method: 354 (M-H). λmax abs 298 nm.

Anti-bactericidal Activity of ZnO Nano-catalyst

ZnO Nano-catalyst was processed and applied to Luria-Bertini (LB) agar plates at varying quantities (10-100 µg/mL) to determine its bactericidal action. After inoculating the plates with E. coli (10^5 CFU), they were incubated for a day at 37 °C.

Using a colony counter, colonies were counted after the incubation period. The ZnO Nano-catalyst was absent from the control plates. There were three duplicates of the experiment run. By adding 100 mL of LB broth medium with 50 µg/mL of ZnO NP, bacteria (105 CFU) were added, establishing the bacterial growth curve. Every 30 minutes, the optical density (O.D.) value was measured at 600 nm to create a growth curve. Without the use of nanoparticles, control was kept constant.

RESULTS AND DISCUSSION

ZnO Nano-catalyst Synthesis and its Characterization

Visual observations of the color shift of the solution from colorless to off-white allowed for the prediction of the production of nanoparticles. Later, UV–visible spectroscopy was used to validate the synthesis of

nanoparticles. The wavelength range in which UV-visible measurements were obtained was 250-500 nm. A significant peak was detected at 340 nm, and the region of 300-370 nm showed the maximum absorption (Fig.-2). The XRD pattern's broad peaks show how the annealing temperature affects increased crystallinity.[23] XRD is based on the Bragg equation $n\lambda = 2d \sin\theta$. The Scherrer equation was used to calculate the crystallite size of the nanoparticles, which was observed between 50 and 80 nm (Fig.-3). The size of the nanoparticles, as determined by the XRD pattern, was further confirmed using a SEM, a microscopic view (Fig.-4), which produced comparable findings. The spherical form of the aggregated nanoparticles was seen in the SEM spectra. The purity of the synthesized nanoparticles was confirmed by the distinct peaks for zinc and oxygen that were clearly visible in the EDAX spectra (Fig.-5). FTIR spectroscopic study discovered the functional groups that acted as a capping agent for the synthesis and stabilization of nano-catalysts, functional groups like phenolic groups, amines, ethers, carboxylic acid, and a hydroxyl group. Strong band shown at 520.63 cm^{-1} resembles Zn-O-Zn stretching vibrations (Fig.-6). After incubating for 10 hours, the ZnO nano-catalyst at a concentration of 50 µg/mL exhibited good suppression of bacterial growth. Twenty hours of incubation resulted in the return of growth in E. Coli, indicating that the nano-catalyst had coagulated and been removed from the LB broth medium following its contact with the interior components of the bacteria (Fig.-7). Bacterial growth is hindered as a result of interactions among positively charged ZnO NPs and the negatively charged bacterial membrane, which compromises membrane integrity and allows intracellular protein leakage.24-27 Using serial dilution, solutions of nano-catalyst were prepared at different concentrations (10-50 µg/mL), and then bacteria were incorporated. The number of established colonies dropped in a concentration-dependent way (Fig.-8). It was discovered that the IC_{50} value was 20 µg/mL.

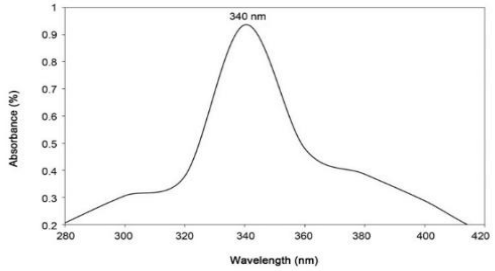


Fig.-2: UV- Visual Spectrum of ZnO Nano-catalyst

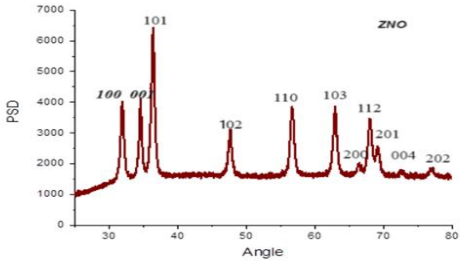


Fig.-3: XRD of ZnO Nano-Catalyst

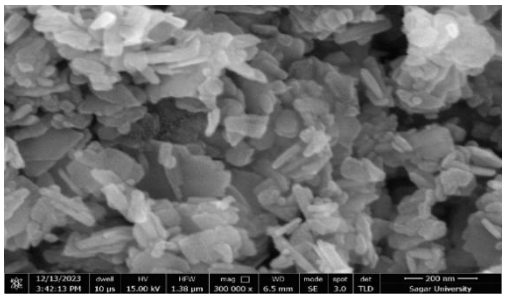
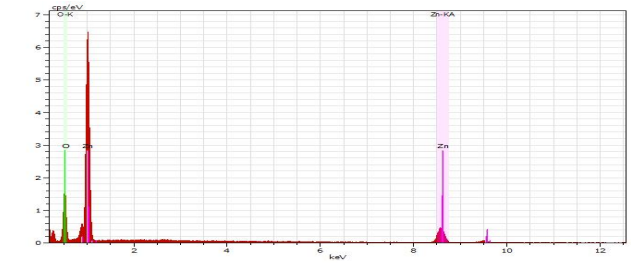


Fig.-4: SEM images of ZnO Nano-Catalyst



El	AN	Series	unn. C [wt.%]	norm. C [wt.%]	Atom. C [at.%]	Error (1 Sigma) [wt.%]
Zn	30	K-series	60.47	76.27	44.02	2.24
O	8	K-series	18.81	23.73	55.98	2.78
		Total:	79.28	100.00	100.00	

Fig.-5: EDAX Spectrum of ZnO Nano-Catalyst

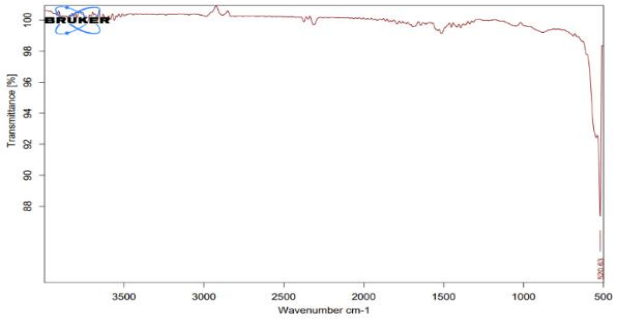


Fig.-6: FT-IR Spectrum of ZnO Nano-Catalyst

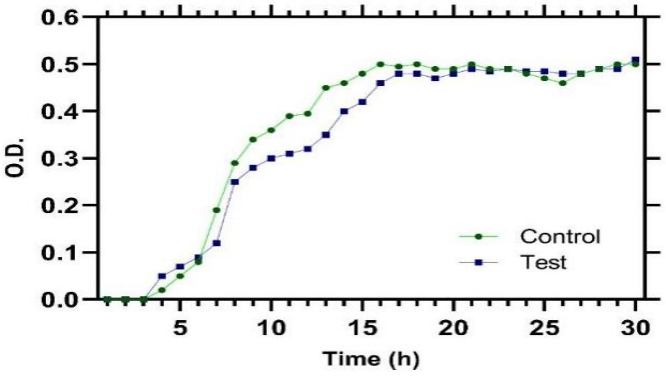


Fig.-7: Bacteria Growth Curve between Test (treated) & Control (native)

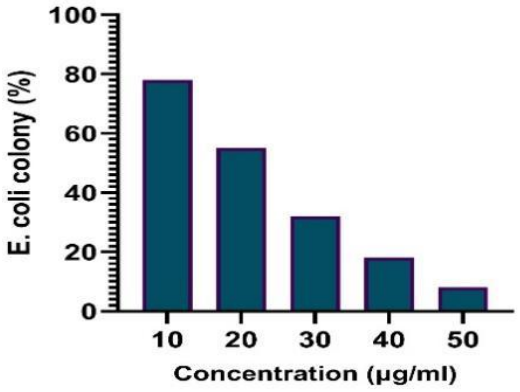


Fig.-8: Anti-bacterial Activity of ZnO Nano-Catalyst

Recyclability of the Nano-Catalyst

The evaluation of a heterogeneous catalyst's recyclability is an essential characteristic that must be examined. Analysis was done on the ZnO nano-catalyst recovery and reusability after the reaction's

completion. The catalyst was separated from the reaction mixture by centrifugation, and before being used again in the next run, it was regularly cleaned three times with hot de-ionized water and acetone to get rid of any organic compounds that had adsorbed. Six successive runs were used to evaluate the catalyst's reusability, and the findings are displayed in Fig.-9. The catalyst was found to be reusable for 6 times with just a little drop-in activity.

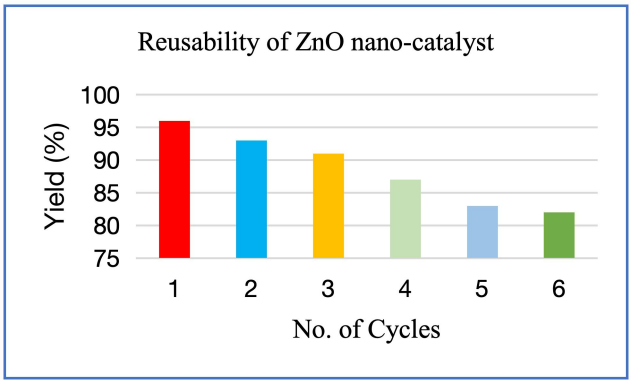


Fig.-9: Recyclability ZnO Nano-Catalyst

We provided a rationale for the successful synthesis of spherical nano-catalysts with an average size range of 1-200 nm. UV-Vis absorption spectroscopy, X-ray diffraction (XRD), Fourier transform infrared spectroscopy (FTIR), and Scanning Electron Microscope in conjunction with Energy Dispersive X-ray investigations (EDX) provide further evidence supporting the size and purity results obtained from the XRD pattern. The antibacterial activity of the ZnO nano-catalyst was shown to be dose-dependent against Escherichia coli (E. coli), with an estimated IC50 value of around 20 µg/mL. This work supports the possible application of ZnO nano-catalyst in several sectors, such as food, pharmaceutical, agriculture, and cosmetic industries, due to its antibacterial properties. We have also employed the obtained nano-catalyst to synthesize derivatives of 1H-indole and (E)-1,3-diphenylprop-2-en-1-one *via* a green approach. NMR, IR, MASS, and UV spectroscopic data are given to characterize the synthesized derivatives.

CONCLUSION

An eco-friendly, quick, easy, non-toxic, and effective method is performed for synthesizing zinc oxide nano-catalyst. Further characterization of the synthesized ZnO nano-catalyst was done by FTIR, XRD, SEM, and UV-Vis absorption spectroscopy. The synthesis of the nano-catalyst was assured by the prominent peak that was detected in the UV-visible spectra at 320 nm. The existence of spherical nano-catalyst with a size range of 60-200 nm was shown by an SEM micrograph. XRD pattern analysis was used to determine the crystallite size even more. The purity of the synthesized nano-catalyst was shown by EDAX and XRD. The distinctive peak for the synthesis of zinc oxide nano-catalyst, shown in FTIR spectra, is located at 520.63 cm^{-1}. This peak resembles the stretching vibration of Zn-O-Zn. By using growth curve analysis and bactericidal activity testing on the ZnO nano-catalyst, this study additionally demonstrated the ZnO nano-catalyst's outstanding antibacterial capability. 20 µg/mL was shown to be the IC_{50} value when bacteria were treated with ZnO nano-catalyst. Notably, ZnO nano-catalysts were used to catalyze the synthesis of 1H-indole-chalcone derivatives via a green approach, mild conditions rationalized high yields above 90 %. The ZnO nano-catalyst is much preferred for organic synthesis, pharmaceuticals, agriculture, and cosmetics.

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CONFLICT OF INTERESTS

The authors declare that there is no conflict of interest.

AUTHOR CONTRIBUTIONS

The present work is related with *SDG-9: Industry, Innovation, and Infrastructure*. All the authors contributed significantly to this manuscript, participated in reviewing/editing and approved the final draft for publication. The research profile of the authors can be verified from their ORCID ids, given below:

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