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Article

Significance of ZnO nano photocatalysts in the clean hospital environment: Effective bacterial disinfection and antibiotic waste (ciprofloxacin) disposal

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Abstract: Hospital waste containing antibiotics is toxic to the ecosystem. Ciprofloxacin is one of the essential, widely used antibiotics and is often detected in water bodies and soil. It is vital to treat these medical wastes, which urge new research towards waste management practices in hospital environments themselves. Ultimately minimizes its impact in the ecosystem and prevents the spread of antibiotic resistance. The present study highlights the decomposition of ciprofloxacin using nano-catalytic ZnO materials by reactive oxygen species (ROS) process. The most effective process to treat the residual antibiotics by the photocatalytic degradation mechanism is explored in this paper. The traditional co-precipitation method was used to prepare zinc oxide nanomaterials. The characterization methods, X-Ray diffraction analysis (XRD), Fourier Transform infrared spectroscopy (FTIR), Ulraviolet-Visible spectroscopy (UV-Vis), Scanning Electron microscopy (SEM) and X-Ray photoelectron spectroscopy (XPS) have done to improve the photocatalytic activity of ZnO materials. The mitigation of ciprofloxacin catalyzed by ZnO nano-photocatalyst was described by pseudo-first-order kinetics and chemical oxygen demand (COD) analysis. In addition, ZnO materials help to prevent bacterial species, S. aureus and E. coli, growth in the environment. This work provides some new insights towards ciprofloxacin degradation in efficient ways.

Keywords: hospital antibiotic waste; ciprofloxacin; ZnO nanomaterials; photocatalytic degradation; bacterial control

1. Introduction

In recent decades, the disposal of hospital harmful wastes, such as antibiotic drugs, directly into municipal wastes has been the major concern to the environment that will affect the animals and human health [1]. The antibiotics in the environment are also a main issue of public concern, since an antibiotic-resistant was formed in exposed non-target organisms [2]. It is urging the development of new guidelines and strategies to detoxify the antibiotic contaminants in the disposal [3]. There are many techniques available for waste treatment, such as advanced oxidation processes, photocatalysis, photolysis, electrolysis, and sonolysis, that are used for the removal of antibiotic toxins [4]. Among various methods, photocatalytic degradation of antibiotic pollutants exhibits low cost and high efficiency. The nanomaterials are serving as an effective photocatalyst for the antibiotic pollutant removal and applications of disinfection in the hospital environment [5].

Nanotechnology plays a great role in the removal of the antibiotic contaminants. Zinc oxide active photocatalysts play an important role in developing environmental challenges such as hospital waste degradation. It paves the way for the efficient oxidation of contaminants under UV or visible irradiation.

Ciprofloxacin is one of the widely used antibiotic medicines to treat infections and antibacterial genes in microorganisms, chosen as a test sample in the present work [6]. The literature revealed that the ciprofloxacin concentration in wastewater treatment plants, raw drinking water, hospital wastewater, lakes, and discharges of the pharmaceutical industry has been reported as 11–99 mg/L, 0.032 mg/L, 150 mg/L, 6.5 mg/L and 31–50 mg/L, respectively. In addition, ciprofloxacin in the hospital environment leads to antibiotic-resistant bacterial formation, and as a result, antibiotic requirements will be higher doses in bacterial infections.

The antibiotic ciprofloxacin with ZnO photocatalyst was tested for finding the degradability of this pharmaceutical pollutant [7]. The present plan is to carry out the UV photocatalysis for the mitigation process, and the usage of ZnO (catalyst) with the different concentrations of antibiotics was discussed in detail [8]. The outcome of the research would meet the great demand of disposal of hospital wastes. Considering the significant detection of ciprofloxacin in hospital environments, the necessity of removing it emerges and leads the way for further large-scale practical applications.

2. Materials and methods

2.1. Synthesis of ZnO Nanoparticles

Zinc acetate ($Zn(CH_3COO)_2(H_2O)_2$) and ammonium hydroxide (NH_4OH) were taken as precursors to prepare ZnO nanoparticles by the co-precipitation. After zinc acetate was completely dissolved in double deionized water, ammonium hydroxide precursor was added dropwise until reaching the *pH* value of 9. The solution was stirred for 3 h at room temperature, and the homogeneity of the solution was attained by continuous agitation using a magnetic stirrer [9]. The resultant white, creamy solution was left to cool down for an hour. The precipitate was washed several times using ethanol and distilled water to reach the neutral value of *pH* and then filtered. Then the precipitate is kept in the muffle furnace at 350 ℃ for 3 h. The steps to prepare ZnO nanoparticles are pictorially shown in **Figure 1** [10].

Figure 1. Steps to synthesize zinc oxide nanoparticles.

2.2. Photocatalytic detoxification of antibiotic contaminants

The photocatalytic antibiotic degradation process was carried out using ZnO (catalyst). The different concentrations of ciprofloxacin solution were prepared by taking 10 mg, 20 mg, 30 mg, 40 mg, and 50 mg dissolved with the distilled water separately [11]. The catalyst (ZnO) of 5 mg is added to the prepared ciprofloxacin solution and stirred at 500 rpm for 30 minutes. After stirring, the solution is exposed to a 254 nm 220V-15W UV lamp in a photo reactor for 30 minutes. The sample of 5 ml can be taken out from the solution of different concentrations, and the absorbance study is analyzed by UV spectroscopy [12].

The degradation capability of catalysts was estimated by finding absorbance with the help of a UV-Vis spectrometer (Shimadzu/UV-2600) and estimated using the formula:

Percentage of dye degradation = [C0–C]/C0 × 100

Here, C0 = [initial adsorption], Ct = [adsorption at time t].

2.3. Results and discussion

Characterization of synthesized ZnO nanoparticle samples

X-ray diffractometer is used to study the phase identification of crystalline material and the structural properties of ZnO. **Figure2(a)** shows the XRD pattern of ZnO nanoparticles. The sharp diffraction peaks of ZnO nanoparticles are observed at 2θ values of 31.68º, 34.10º, 36.49º, 47.41º, 56.81º, 66.36º, 67.98º, 69.27º and 77. 55º is associated with the (100), (002), (101), (110), (103), (200), (112), (201) and (202) planes respectively. These peaks correspond to the phase with hexagonal crystal geometry (JCPSD card No. 01-007-2551). The observed diffraction peaks are consistent with the reported values, and the absence of any peaks associated with impurities indicates the purity of this phase [13].

The crystallite size of nanoparticles is calculated by using the Debye-Scherrer formula.

$$\mathrm{D} = \frac{k\lambda}{\hat{a}\ \cos\theta}$$

where, D—crystallite size, k—shape factor (0.9), λ—Wavelength of the X-ray source, β—Full-width half maximum height, θ—is the angle of diffraction [14]. The maximum intensity of ZnO nanoparticles occurs at 2θ = 36.49º is associated with (101) plane. The average crystallite size of ZnO nanoparticle is calculated as 23.38 nm.

The FTIR spectrum was recorded using Perkin Elmer FTIR spectrometer in the range at 400–4000 cm^{-1}. Fourier transform infrared spectrum of ZnO is shown in **Figure 2b**. The observed absorption peaks are 3439 cm^{-1}, 1708 cm^{-1}, 1643 cm^{-1}, 1449 cm^{-1},1027 cm^{-1}, 863 cm^{-1}, 767 $cm^{-1}$and 608 cm^{-1} [15]. The typical peaks at 608 cm^{-1}, 767 cm^{-1} and 1027 cm^{-1} related to stretching vibrations of Zn–O bonds. The absorption at 863 cm^{-1} is due to the formation of tetrahedral coordination of Zn. The peak at 3439 cm^{-1} corresponds to the stretching vibration of the –OH molecule, while the band at 1643 cm^{-1} shows the effect of the bending vibration of H–O–H bond a water molecule and is also observed in the significant absorption peak at

1449 cm^{-1}. The peak at 1708 cm^{-1} can also be related to an additional common band attributed to the bending mode of absorbed water.

The optical absorption of ZnO nanoparticle samples was determined at room temperature using the UV-visible spectrophotometer in the range of 100–1400 nm. **Figure 2c** shows the absorption spectrum of ZnO, which exhibits an absorption edge in the range of 376 nm, which relates to the UV region. The outcome reveals that there is a strong absorption in the UV band and weak absorbance in the visible region. Therefore, these ZnO samples can be used as an efficient photocatalyst under irradiation with UV light of wavelength less than 400 nm [16].

Figure 2d is the SEM image of ZnO nanoparticles, captured under 10.00 KX magnifications. It is predicted from the SEM image that ZnO nanoparticles are homogeneously scattered, and also the randomly oriented hexagonal shapes of nanosized particles with uniform distribution are presented [16]. The exhibited agglomeration of hexagonal-shaped nanoparticles is correlated with the outcome of X-ray diffraction analysis.

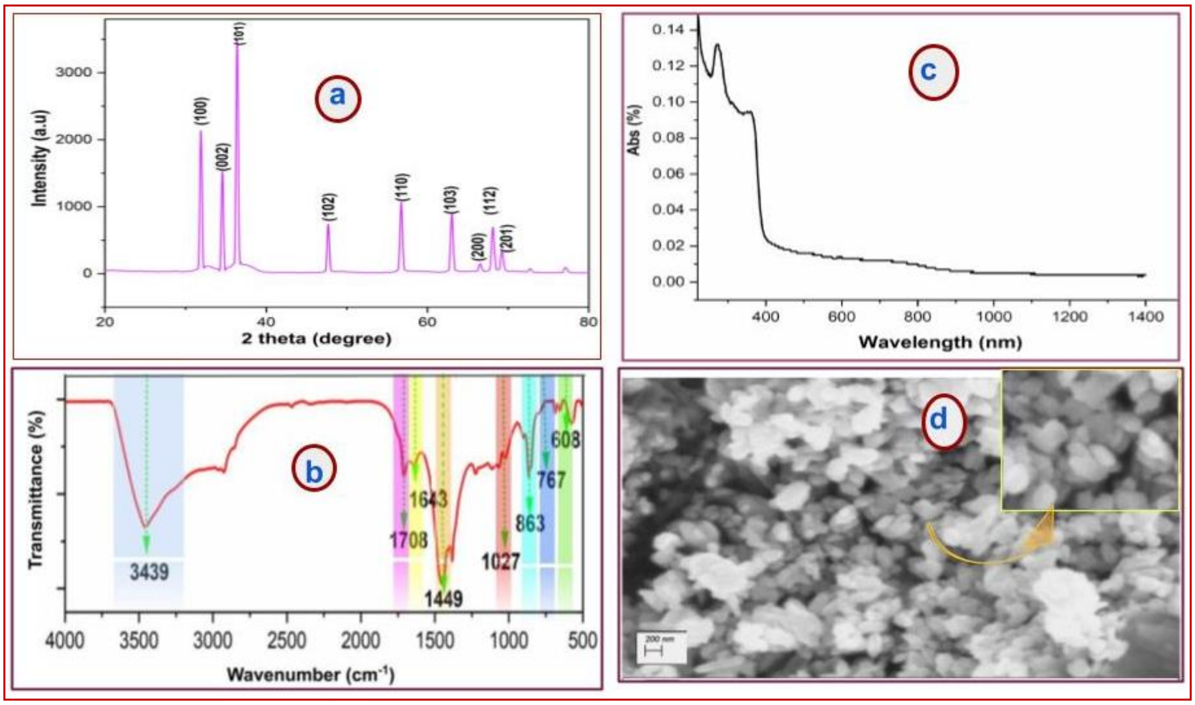

Figure 2. (a) X-Ray diffraction; **(b)** FTIR vibrations; **(c)** UV-Vis absorption; **(d)** SEM micrograph characterization of zinc oxide nanoparticles.

3. Applications

3.1. Antibacterial activity

The bacterial strains of Staphylococcus aurous—902 (gram—positive species) and Escherichia coli—443 (gram—negative species) for agar well diffusion was purchased from MTCC, Chandigarh, India and the antibacterial activity of ZnO nanoparticles was tested against these bacterial strains [17]. Petri plates containing 20 ml nutrient agar medium seeded with the bacterial strains were cultured for 24 h, and adjusted to 0.5 OD value according to MCFarland standard (Staphylococcus aurous—902 and Escherichia coli—443).

Figure 3 shows the synthesized ZnO materials exhibit zones against pathogenic bacteria in the petri plates. The diameter of the zone is measured in millimeters and also exhibits the degree of susceptibility of microorganisms. The zones of inhibition of ZnO nanoparticles are (14.91 ± 0.16) mm and (17.25 ± 0.35) mm for E.Coli and S.aureus respectively, as comparable with the standard values of positive control (E.Coli:16.75 ± 0.06, S.aureus: 17.25± 0.21 in mm). The result proves that the effective antibacterial activity of ZnO samples.

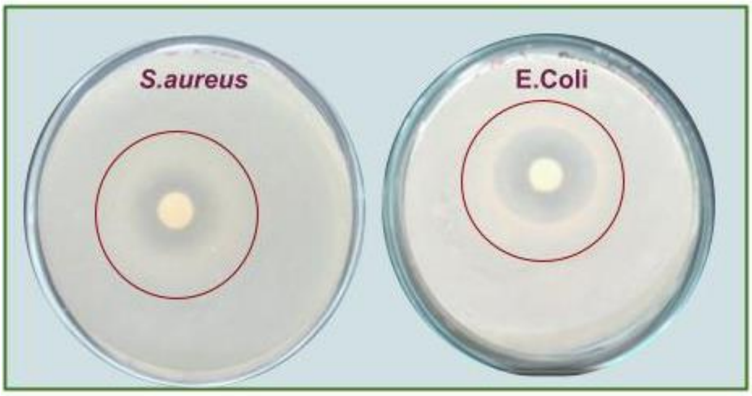

Figure 3. Antibacterial activities of the ZnO nanoparticles.

3.2. Photocatalytic degradation mechanisms of ciprofloxacin

(Figure 4a–d) depicts When the ultraviolet light was irradiated on the ZnO semiconducting nanomaterial, the valence band electrons absorbed the energy and shifted to the conduction band. As a response, charged particles (holes in the valence band and electrons in the conduction band) develop [18]. The excitation of electrons from the valence band to the conduction band of ZnO catalyst due to UV illumination and the excitation process continuously produces the electron-hole pairs. Following that, the antibiotic waste components interact directly with the released charged particles, either degrading them or transferring them to the surface of the ZnO semiconductor photo catalyst. As the active sites are decreased, the compound is greatly absorbed by the catalyst. ZnO possesses high photocatalytic performance, well-separated reductive and oxidative active sites, generation of reactive oxygen species (ROS), and strong redox ability [19]. This reduces the reaction between the holes and the electrons. When the holes (h^+) are reacted with the water molecules to form hydroxyl free radicals ($OH^.$) and the electrons ($e^−$) are reacted with dissolved oxygen to produce superoxide free radicals ($O_2^{.−}$). Free radicals such as ($OH^.$) and ($O_2^{.−}$) are the end products [20]. These two products ($OH^.$) and ($O_2^{.−}$) are strong oxidizing agents and are reacted with the ciprofloxacin to degrade it as a simple substance such as CO_2, H_2O and Mineral acids.

The maximum absorbance of ciprofloxacin is obtained at the wavelength of 272 nm [21]. The elimination rate of an antibiotic is greatly improved by considering low initial concentration. From the study, the least concentration of antibiotics achieves a high removal rate that is due to more reactions taking place for a lower number of antibiotic molecules [22]. The effect of different concentrations of the antibiotic ciprofloxacin treated with ZnO nanoparticles and the degradation plot is shown in **Figure 4(a)**.

The mechanisms are expressed in Equations (1)–(4).

$$ZnO + hv \rightarrow ZnO(e^- + h^+) \quad (1)$$

$$e^- + O_2 \rightarrow O_2{}^{\cdot -} \quad (2)$$

$$h^+ + H_2O \rightarrow OH^{\cdot} \quad (3)$$

$$OH^{\cdot} + O_2{}^{\cdot -} + ciprofloxacin \rightarrow CO_2 + H_2O + mineral\ acid \quad (4)$$

The antibiotic ciprofloxacin is degraded and exposed to subsequent processes, resulting in non-toxic chemicals. When the polluting substance is present in higher concentrations, a smaller number of photons only penetrate through the surface catalyst that reduces the degradation studies.

- Kinetic model

The kinetic model indicates the photo degradation of contaminants fits well with the pseudo-first-order kinetic plot. The high R2 values (more than 0.9) of the linear plot obey first-order reaction kinetics, supporting that the degradation of ciprofloxacin degradation using ZnO [23]. **Figure 4b** has the pseudo-first-order rate constants of pollutants at 0.0507 min^{-1}.

- Recycling and reusage

After the reaction is completed using a simple centrifugation method, ZnO nanoparticles were separated and dried at room temperature. Then the possibility of recycling of the used samples was analyzed for further degradation of antibiotic ciprofloxacin contaminants. ZnO nano-catalyst was tested for its photocatalytic degradability up to four cycles and retains its stability as in **Figure 4c**. Hence its reusability, it is recommended that ZnO might be a suitable photocatalyst for degrading antibiotic molecules [24].

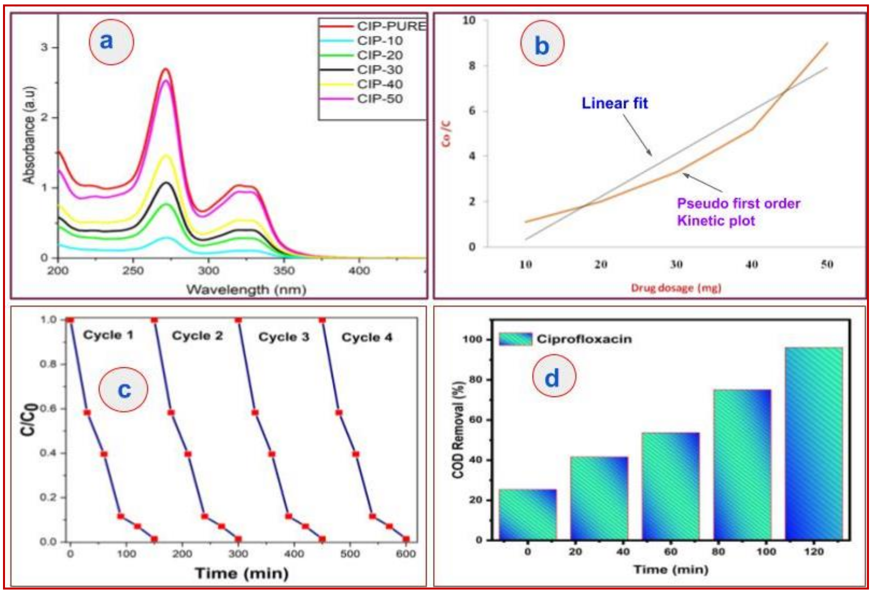

Figure 4. UV Absorbance of effect of different concentration of antibiotic ciprofloxacin.

3.2.1. Chemical oxygen demand (COD) analysis

The chemical oxygen demand (COD) test is used to determine the natural chemicals in a solution that are prone to pollutant degradation due to light illumination. A higher COD suggests the presence of more organic contaminants in the environment. In our study, the required quantity of chemical oxygen was calculated for the photocatalytic breakdown of antibiotics in hospital waste as an outcome of ultraviolet light exposure. Oxidation of organic molecules produced CO_2. In addition to H_2O, electrons were generated, the oxidizing reagent was reduced, and the liberated electrons were embraced. The COD of the solution was evaluated using the formula below to determine photo catalytic efficiency [(% = (COD0–CODt). 100%/COD0 where COD0 and CODt] are the (COD0–CODt) and COD values of the solution before photodegradation and during this process at a given time [25]. **Figure 4d** shows the results in (22.5%) chemical oxygen demand eradication, which equates to (80%) decomposition process. While decomposing ciprofloxacin, hydroxyl radicals are more readily released; this results in the photocatalytic degradability. This indicates that the photocatalytic process is quite effective in removing the ciprofloxacin from the solution.

3.2.2. X-Ray photoelectron spectroscopy

X-ray photoelectron spectroscopy (XPS) was taken for ZnO samples to illustrate their electronic states and surface materials. XPS describes the elemental bonding, chemical composition, and oxidation states in the environments of ZnO surfaces. **Figure 5** confirmed the the surface compositions and related valence states of the high-resolution spectra of Zn 2p. The Zn $2p_{3/2}$ and Zn $2p_{1/2}$ of Zn^{2+} are responsible for the peaks centered at 1022.2 and 1045.3 eV, respectively [26]. XPS characterization results of the catalyst before and after the reaction indicate that the presence of only Zn^{2+}. Furthermore, the XPS spectra confirmed that ZnO is well-formed and the purity of the samples was retained.

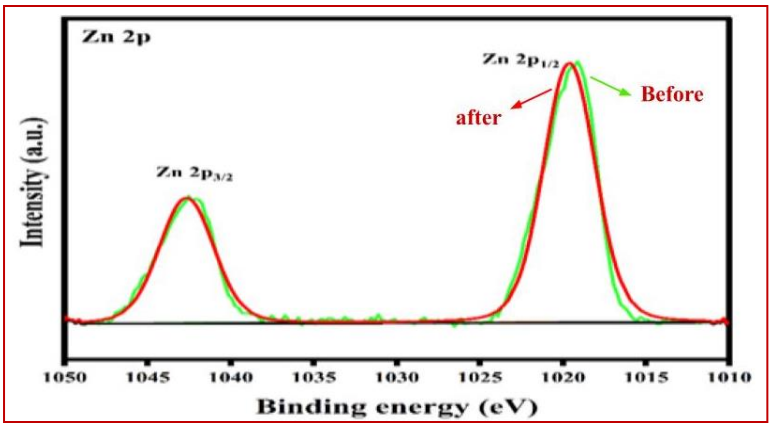

Figure 5. XPS plots of ZnO samples before and after usage for photo degradation.

4. Conclusion

The photo catalyst ZnO is a potent nonmaterial for hospital drug waste treatment. ZnO nano materials were prepared by the co-precipitation chemical method. The samples purity, crystalline nature and crystallite size were studied using

X-ray diffraction analysis. The functional groups of prepared samples were confirmed by FTIR technique. UV-V is analysis confirmed its suitability as an efficient photo catalyst for the removal of the antibiotic ciprofloxacin in the wastewater. SEM photo images revealed the homogeneous growth of hexagonally edged ZnO nanoparticles, and the crystallite sizes are 20 nm to 30 nm comparable to the calculated value of 23.38 nm by Debye-Scherer in XRD. The effective photocatalytic degradation of ciprofloxacin antibiotic was achieved in the presence of ultraviolet light illumination. The highest degradation was found in the lowest concentration of the antibiotic (10 mg). ZnO mixed with ciprofloxacin is against *S. aurous* (gram—positive species) and *E. coli* (gram—negative species) bacterial strains. Therefore, this can be concluded that the proposed nano photocatalysis is an economic, low-time-consuming, and good photocatalytic degradation takes place with the antibiotic ciprofloxacin.

Highlights

- ZnO nanoparticles are chosen as an efficient photocatalyst for ROS generation.
- Effectively decompose the hazardous ciprofloxacin (test sample) in hospital waste.
- Efficient antibacterial agent to control *S. aurous* and *E. coli* growth.

Author contributions: Investigation, JBN; resources, JBN; data curation, JBN and PM; Writing—original draft preparation, JBN and PM; antibiotic—ciprofloxacin and antibacterial activity, JBN; experimental work, PM; conceptualization, JRMB; project administration, JRMB; supervision, JRMB; validation, JRMB; visualization, JRMB; writing—review and editing, JRMB. All authors have read and agreed to the published version of the manuscript.

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