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Synthesis, Antimicrobial Activity, and Photocatalytic Performance of Ce Doped SnO_2 Nanoparticles

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This work represented the synthesis of Ce doped SnO_2 nanoparticles by a wet chemical method and was characterized by various characterization techniques. PXRD confirmed the presence of the rutile phase for Ce doped SnO_2 nanoparticles. SEM image and elemental mapping showed agglomerated irregular shaped particles and uniform distribution of 5% Ce ions within the SnO_2 lattice, respectively. Ce doped SnO_2 nanoparticles showed antimicrobial activity against *E. coli* and prevented the growth of bacteria. The nanoparticles were found photocatalytic active and photocatalytic behavior was elucidated by the degradation of Malachite Green dye under UV light irradiation.

Keywords: cerium, *E. coli*, malachite green, photocatalyst, doping

INTRODUCTION

There has been a continuous threat to health, food packaging, cosmetics, and many more industries due to microbes and these industries highly depend on various antimicrobial agents (Ananpattarachai et al., 2009). Contaminated surfaces, colonization, subsequent biofilm formation, improper cleaning of equipments are found to be the primary carriers of microorganisms leading to several foodborne and other outbreaks (Swaminathan and Smidt, 2007; Yemmireddy and Hung, 2017). Nanotechnology has its impacts on all fields of science related to nanomedicine, biomedical, biosensor, development of smart cities, energy, environment, etc. (Kumar et al., 2019a; Bhawna et al., 2020; Gupta et al., 2020a,b).

Nanoparticles have been long known for their antimicrobial behavior against gram-positive and gram-negative bacteria, pathogens, and other microbes (Azam et al., 2012; Vargas-Reus et al., 2012). Metal oxide nanoparticles serve as antimicrobial agents owing to their large surface area (Raghunath and Perumal, 2017). Out of several metal oxide nanomaterials, scientists have more interest in SnO_2 nanoparticles because of their novel properties such as high chemical stability, high transparency, and low electrical sheet resistance, etc. (Jarzebski and Marton, 1976a,b; Jarzebski and Morton, 1976). The modified SnO_2 also has great technical and scientific interests because of its diverse applications, e.g., transparent conducting electrodes, gas sensors, as electrodes in lithium-ion batteries, electronic devices, dye-based solar cells, H_2 generation, etc. (Jiang et al., 2017, 2018; Park et al., 2017; Xie et al., 2017; Wang et al., 2018; Bhawna et al., 2020). Other than these applications, SnO_2 has been seeking attention as an antimicrobial agent and has played an essential role against the growth of various bacterial strains like *Staphylococcus aureus*, *E. coli*

(Kumari and Philip, 2015; Vidhu and Philip, 2015; Phukan et al., 2017). Green synthesis of SnO_2 nanoparticles using *Aloe barbadensis miller* showed antibacterial and antifungal activities (Ayeshamariam et al., 2013). Apart from antimicrobial activities, other important biological properties like anticancer, antitumor, and antioxidant activities have also been reported using green synthesized SnO_2 nanoparticles (Kamaraj et al., 2014; Khan et al., 2018). However, when doped with transition metal ions, SnO_2 disinfects microbes with good efficiency, i.e., Co-doped SnO_2 and Ag-doped SnO_2 nanoparticles have shown potent antibacterial activities (Chandran et al., 2015; Nasir et al., 2017; Qamar et al., 2017; Ali et al., 2018). Only a few reports are available on antimicrobial activities of Ce doped metal oxide NPs. Ce-doped ZnO NPs showed antimicrobial activity against both gram-negative and gram-positive bacteria (Rooshde et al., 2020). Similarly, Ce doped CuO NPs completely eradicated the *E. coli* and *S. aureus* bacteria (Jan et al., 2014) and Ce-doped ZrO_2 NPs showed high antibacterial property against gram-positive bacteria than gram-negative bacteria (Mekala et al., 2018).

Water is a crucial factor for the existence of life on earth and clean water is the necessity of the hour. Consumption of water by a rapid increasing population is leading to the depletion of major aquifers. On the other hand, organic manufacturing industries have been the target for disposing of their chemical wastes into water bodies. According to research, the world's dye production of about 0.7 million tons (>11%) is annually released as industrial wastewater (Samadi et al., 2019). Among various known dyes, Malachite green dye has its extensive uses worldwide. Besides its use as a dye in silk, jute, leather, wool and paper industries; it is also used as a food additive, coloring agent and as a disinfectant. However, due to its carcinogenic effects on human health and aquatic life, it has now become a controversial compound and has been banned in many countries. Continuous efforts are being made to recycle contaminated water containing bacteria, toxic chemicals, dyes, heavy metals, etc. to make it safe for drinking and other purposes. Some conventional methods are- photocatalysis, ozonation, Fenton's reagent, electrochemical routes, membrane filtration, coagulation, adsorption, ion-exchange, irradiation, anaerobic and aerobic degradation, etc. (Gusain et al., 2019). Though, metal oxides such as TiO_2, SnO_2, ZnO have been found as better photocatalysts for the degradation of organic dyes in aquoues solution. SnO_2 as an n-type semiconductor has also been reported for the degradation of various azo dyes. Besides antimicrobial activities, doped SnO_2 finds improved results in photocatalytic activities. Ce doping has been known for bandgap tailoring as well as lattice distortion in SnO_2. There are various methods reported in literature for the synthesis of Ce doped SnO_2 such as- sol-gel (Shide et al., 2010; Ahmed et al., 2019), hydrothermal (Lian et al., 2017), co-precipitation (Bharathi et al., 2017; Kumar et al., 2018), wet-chemical (Kumar et al., 2019b), flame spray method (Kotchasak et al., 2018), etc. Kumar et al. showed degradation of dyes such as methylene blue and methyl orange using Ce doped SnO_2 nanoparticles (Kumar et al., 2019b) whereas Wu et al. degraded methyl orange dye using Ce doped SnO_2 (Shide et al., 2010).

To the best of our knowledge, until now, no work has been reported on antimicrobial behavior using Ce doped SnO_2. This work involves the facile synthesis of Ce doped SnO_2 nanoparticles and reports its antimicrobial behavior against microbes. It also represents photocatalytic degradation of malachite green dye using Ce doped SnO_2.

EXPERIMENTAL SECTION

Ce doped SnO_2 nanoparticles were synthesized by a wet-chemical method using hydrogen peroxide, as mentioned in our previous report (Kumar et al., 2019b). Solutions of $SnCl_2.2H_2O$ (Merck, 18 ml of 0.5 M) and $CeCl_3$ (Merck, 6 mL of 40 mM) were mixed and 30 mL hydrogen peroxide was added into the mixture. Then, the mixture was refluxed at 100°C for 14 h. The white suspension was cooled to room temperature, centrifuged, and was dried after washing several times to remove dissolved impurities.

Characterization Details

The powder X-ray diffraction (PXRD) pattern was recorded using Rigaku, Miniflex 600 X-ray diffractometer employing monochromatized Cu K_α radiation. The Field Emission Scanning Electron Microscope (FESEM) image of the SnO_2 NPs was recorded on a ZEISS Gemini SEM 500.

EVALUATION OF ANTIMICROBIAL ACTIVITY

The Antimicrobial activity of Ce doped SnO_2 nanoparticles was carried out using Diffusion Susceptibility Test method (Bauer et al., 1966). The bacterial strain, *E. coli* was inoculated in 5 ml LB Media (Luria-Bertani; HiMedia Laboratories) and was kept at 37°C and 180 rpm for overnight incubation. The overnight incubated bacterial culture was diluted in 1:100 ratios with fresh LB media. A zone of inhibition experiment was analyzed using an LB Agar plate well-diffusion method. Then, the sterilized

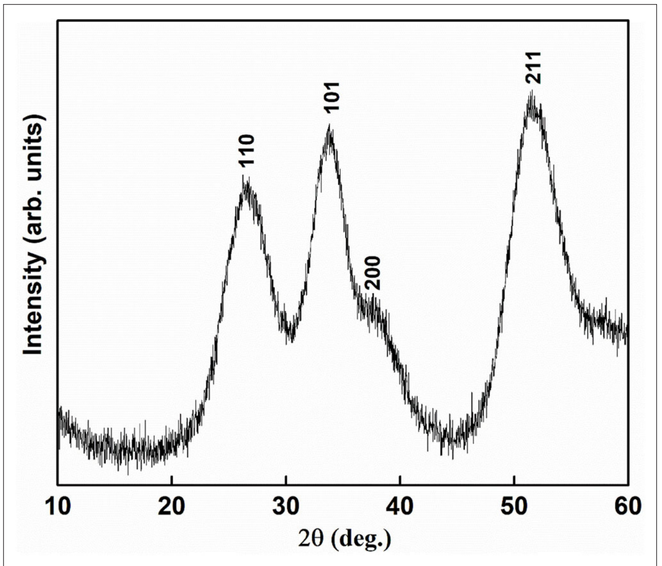

FIGURE 1 | PXRD pattern of Ce doped SnO_2.

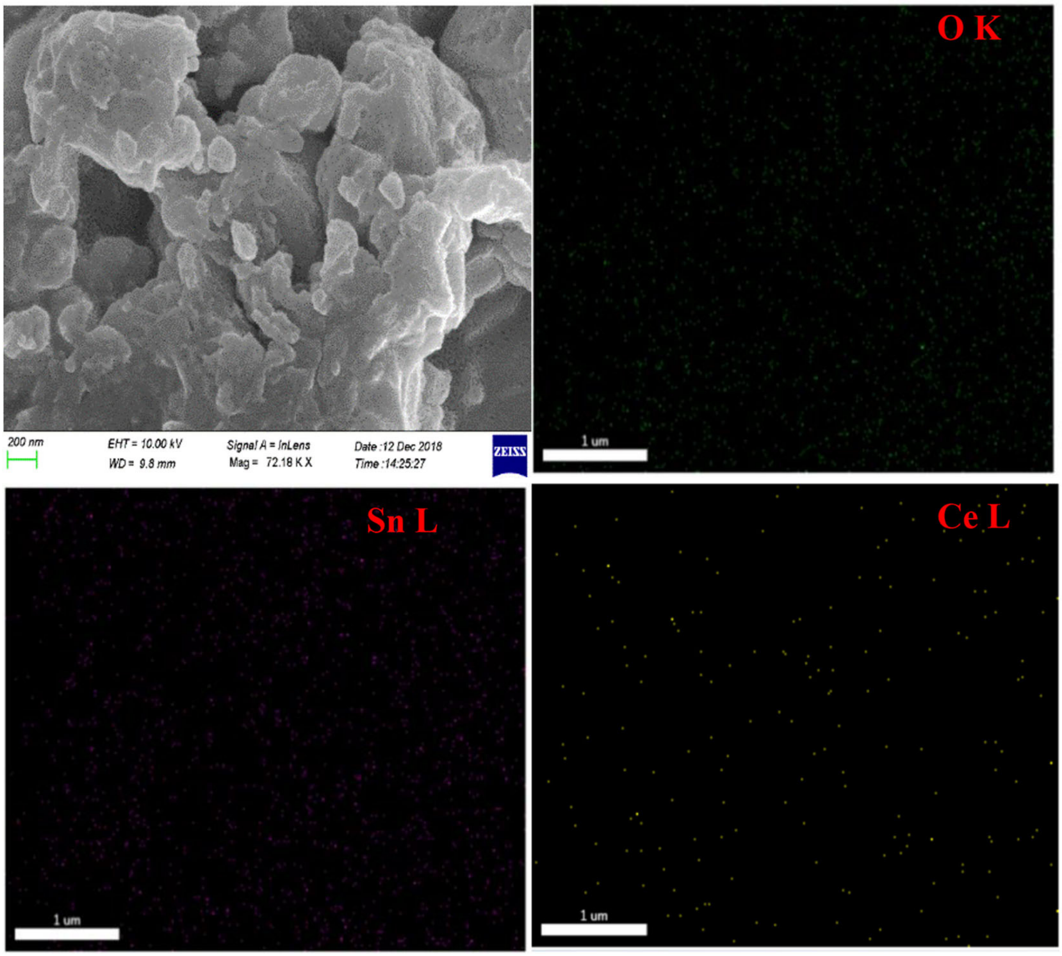

FIGURE 2 | SEM image and Elemental mapping of Ce doped SnO_2.

well cutter was used for boring the LB Agar plate. The diluted overnight bacterial culture of *E. coli* was spread on LB agar plate. Thereafter, seven concentrations of Ce: SnO_2 NPs, namely, 0.25, 0.50, 1, 2, 3, 4, and 5 mg were poured into LB Agar wells. The well in the center of LB agar plate did not contain Ce doped SnO_2 NPs and was used as a control. Thereafter, the LB agar plate with Ce doped SnO_2 NP was incubated at 37°C for 16 h.

Photocatalytic Degradation of Pollutants

Photocatalytic degradation of dye was performed in an in-house fabricated solar reactor under UV ($\lambda < 400$ nm) light by high vapor pressure mercury lamp 125 W (Osram, India) (Kumar et al., 2011). In the photocatalytic activity, 0.1 g of Ce: SnO_2 NPs were suspended into an aqueous solution of 100 mL of 15 μM MG dye, which was taken in the photoreactor. The dye solution suspended with the catalyst was stirred for 30 min in the dark to attain the equilibrium and then the light was irradiated over the solution. Each time, five mL volume were pipetted out timely, centrifuged and the absorbance was noted using the UV-visible spectrometer.

RESULT AND DISCUSSION

The Powder X-ray diffraction pattern of the synthesized Ce doped SnO_2 nanoparticles has been shown in the **Figure 1**. It shows a rutile structure with tetragonal symmetry space group $P4_2/mnm$ [a = 4.680 (4) Å and c = 3.167 (4) Å] and shows clear reflection at (110), (101), (200), and (211) crystallographic planes corresponding to JCPDS file no. 41-1445 (Kumar et al., 2019b). The absence of any other characteristic peaks rule out possibilities of impurities or other species within the lattice represents high phase purity. The broadness of the diffracted peaks depicts a small size of crystals and the average crystallite size determined using the Scherrer formula was found to be ~6 nm (Scherrer, 1912).

The morphology and elemental mapping of Ce doped SnO_2 nanoparticles was investigated through FESEM (**Figure 2**). Irregularly shaped particles distributed unevenly over the lattice surface has been shown through SEM imaging. Elemental mapping shows the spatial distribution of elements within the lattice and provides the evidence that Ce (yellow), Sn (purple), and O (green) were homogeneously distributed within the crystal lattice.

After elucidation of the phase formation and morphology of the formed nanoparticles, the concentration of Ce ions was found to be 5% as determined through X-ray photoelectron spectroscopy in our previous report (Kumar et al., 2019b). Also, the presence of Ce^{3+} and Ce^{4+} ions was confirmed into SnO_2 lattice, which caused charge imbalance and hence disorderness in the lattice (Kumar et al., 2019b).

Analysis of Antimicrobial Activity

Antibacterial activity of Ce doped SnO_2 NPs was observed on LB Agar well-diffusion method (Dil and Sadeghi, 2018). Antibacterial activity of the NPs was compared with the control well (without NPs). The antibacterial activity of Ce doped SnO_2 NPs was not observed at concentration 0.25–3 mg (**Figure 3**). **Figure 3** suggested that the zones of inhibition were prominent at two concentrations namely, 4 and 5 mg, respectively and was highest at 5 mg concentration. The antibacterial activities have been assessed through the diameter of the zone of inhibition. At a concentration of 4 mg or above, Ce doped SnO_2 NPs showed potent antibacterial activities (**Table 1**). Previously, other metal ions doped with SnO_2 like Co-doped SnO_2, Cu-doped SnO_2, Fe-doped SnO_2, and Ag-doped SnO_2 nanoparticles have also been reported for their antibacterial activities (Chandran et al., 2015; Nasir et al., 2017; Ali et al., 2018; Baig et al., 2020; Sathishkumar and Geethalakshmi, 2020). Generally, nanoparticles kill the bacteria through cell membrane disruption, free radical formation causing reactive oxygen species responsible for antibacterial action (Sirelkhatim et al., 2015).

Photocatalytic Dye Degradation

The degradation of malachite green was performed photocatalytically using Ce: SnO_2 nanoparticles (**Figure 4**) under UV light irradiation. It degrades malachite green dye ~50% in 120 min of light irradiation. When compared with other metal oxides, it is found that undoped TiO_2 NPs and F doped TiO_2 NPs photocatalytically degraded 99.9 and 54.26% MG dye in 240 min and 120 min, respectively (Chen et al., 2007; Panahian and Arsalani, 2017). Sn doped TiO_2 has been reported degrading 85% MG in 340 min under light irradiation (Sayilkan et al., 2007), while SnO_2 NPs degraded 27% MG in 180 min under UV light irradiation (Kumar et al., 2016).

The probable mechanism for the degradation of malachite green dye using Ce doped SnO_2 NPs has been revealed in **Figure 5**. Electrons were excited into the conduction band of Ce doped SnO_2 nanoparticles from its valence band on light irradiation [bandgap = 3.80 eV (Kumar et al., 2019b)]. Electrons were also injected into the conduction band of photocatalyst after transfer from HOMO to LUMO of malachite green dye (Helaïli et al., 2017). These electrons from two different sites then move to the surface for surface reactions. The electrons at the surface react with adsorbed/dissolved oxygen to produce $\dot{O}_2^-$ radical. The concentration of the O_2 molecule is responsible for the efficiency

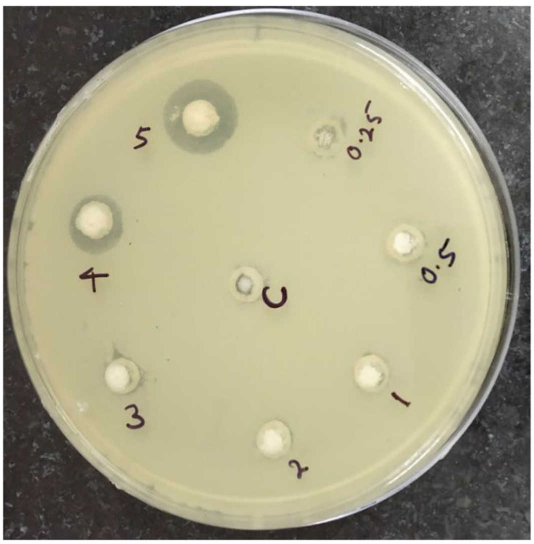

FIGURE 3 | Antibacterial activity of Ce-doped-SnO_2 NPs.

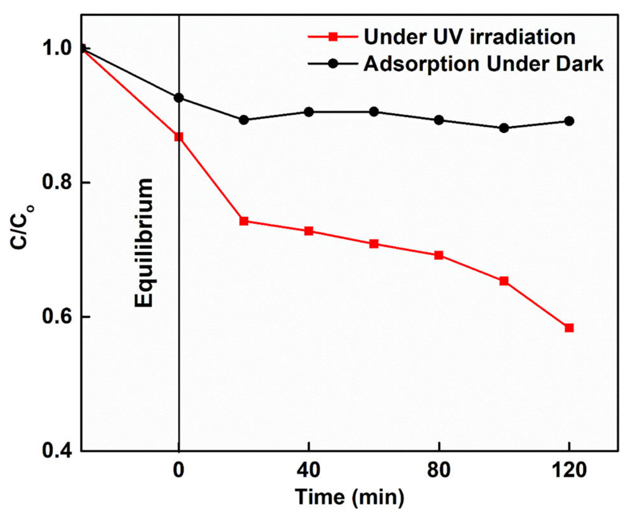

FIGURE 4 | Adsorption of MG in dark and photocatalytic degradation of MG in the presence of Ce doped SnO_2 under UV irradiation.

TABLE 1 | Concentration and observation of zone of inhibition of Ce doped SnO_2.

Concentration of Ce doped SnO_2 (mg)	0.25	0.50	1	2	3	4	5
Zone of inhibition	No	No	No	No	Least	Moderate	Strong activity

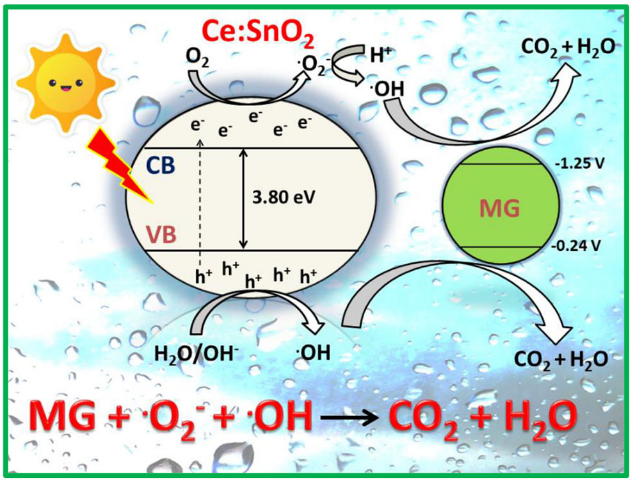

FIGURE 5 | Proposed malachite green degradation mechanism using Ce doped SnO_2 nanoparticles.

of degradation as these molecules scavenge the electrons in the conduction band, preventing electron-hole recombination. Moreover, holes in the valence band react with water molecules or hydroxide ions to produce hydroxyl radicals ($OH^{\cdot}$) (Kumar et al., 2016; Ma et al., 2018). The generated oxidizing agents (superoxide radical anions and hydroxyl radicals) contributed to the oxidative degradation of malachite green, which was then converted into simple and less harmful products. The high stability of Ce doped SnO_2 NPs mentioned in the previous report and hence, these nanoparticles can be reused without undergoing any change in structure (Kumar et al., 2019b).

CONCLUSION

Facile and economical synthesis of Ce doped SnO_2 NPs showed potent antimicrobial properties so far. Also, nanomaterials were able to degrade toxic organic pollutants like malachite green. These nanomaterials could be used against bacterial infection as well as for multidrug-resistant bacteria along with wastewater treatment purposes.

DATA AVAILABILITY STATEMENT

The raw data supporting the conclusions of this article will be made available by the authors, without undue reservation.

AUTHOR CONTRIBUTIONS

All authors listed have made a substantial, direct and intellectual contribution to the work, and approved it for publication.

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