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Synthesis, Characterization and Antimicrobial activity of Sm^{3+} doped TiO_2 Nanoparticles

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ABSTRACT: Pure and Sm^{3+} ions doped TiO_2 nanoparticles were prepared by low temperature solution combustion method, with Glycine and Ammonium acetate as fuels. The nanoparticles obtained were subjected to PXRD studies for purity and shape. The antimicrobial activity of the nanoparticles on *E.coli* a Gram negative bacterium was investigated by using pure and doped TiO_2 by Disc and Kirby-Bauer method. The studies revealed increased and elevated bactericidal activity of doped TiO_2 in comparison to the pure. The observation was confirmed by the Colony forming units on LB agar, Growth measurement curve by turbidity technique LB broth.

Keywords: XRD, *E.coli*, *Gram –bacilli*, LB broth, Bactericidal activity, Colony forming units.

INTRODUCTION

The synthesis of nano sized particles of TiO_2 doped Sm^{3+} ions with antibacterial properties is of great interest for the development of new biomedical applications. The adaptation of bacteria and resistance to wide range of antibiotics has led to the emergence of some different infectious diseases. The treatment towards these diseases is becoming a difficult task. Hence an effort was made to study. The aim of this study was to evaluate TiO_2: Sm^{3+} nanoparticles for their antimicrobial activity against E.coli, a Gram –ve Bacteria. The fuels used were Glycine and Ammonium acetate for combustion.

2. MATERIALS AND METHODS

2.1 Materials

LB broth, LB agar, Petri plates, Pipettes, Micropipettes, Vials, Double distilled water, *E.coli* culture, *Bacillus* culture, Samarium, Titanium dioxide, UV trans illuminator, Conical flask, Alcohol, Beaker, Streptomycin, Sterilized discs, Sterilized swabs, Cork borer, Incubator[37°C], EMB agar, Nutrient agar, Sonicator, Laminar air flow, Water bath, Oven, Muffle furnace, Methanol, Sulphuric acid, Hydrochloric acid, Autoclave, Glycine, Ammonium acetate, Nitric acid (Chemicals used were of analytical grade). Luria bertani broth, Luria bertani agar, Muller Hinton agar, Nutrient agar, Eosin Methylene Blue Agar were used.

2.2 Method of Preparation TiO_2: Sm^{3+} (0.25-0.75 mol %)

The pure and Sm^{3+} ions doped TiO_2 NPs were prepared from Titanium IV butoxide and samarium nitrate using glycine and ammonium acetate as fuels for low temperature solution combustion method using the stoichiometric calculations. The pre heated muffle furnace at 500^0C was used for combustion of the uniformly mixed solutions using magnetic stirrer. Flakes obtained are ground to form powder and the same is calcinated at 8000C for 2 hours to remove the impurities present in the same. The same procedure was repeated to get the Sm^{3+} doped TiO_2. The antimicrobial activity was done by serially diluting the sample in distilled water and filling into the wells made in the LB agar media. The results are observed after 24H of duration.

2.3 Preparation of stock solution

Stock solution of TiO_2 NPs (both pure and doped) with concentration of 1 mg/ml was prepared and suspended in distilled water. The solution was sonicated for 5 minutes to get a homogenous suspension. The suspension was exposed to UV rays for 30 minutes for the nanoparticles activation.

Table 1: preparation of stock solution

Stock (mg/ml)	0.2	0.4	0.6	0.8	1.0
Distilled water(mg/ml)	0.8	0.6	0.4	0.2	0.0

2.4 Disc method

E.coli inoculated by lawn culture on LB agar. The sterilized whatman paper discs were dipped in

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pure TiO_2 nanoparticles of concentration 0.2, 0.4, 0.6, 0.8 and 1.0 mg/ml and doped TiO_2 nanoparticles of different mole percentage (1%, 3%, 5%, 7%, 9%) of concentration 0.2, 0.4, 0.6, 0.8 and 1.0 mg/ml were placed on the agar surface and it was labeled. Two plates were kept as control using streptomycin, the discs were dipped with streptomycin of concentration 0.2, 0.4, 0.6, 0.8 and 1.0 mg/ml for *E.coli*. These plates were allowed to dry and then kept for incubation at 37^0c for 24 hours. The zone of inhibition was observed.

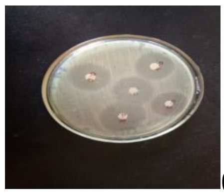
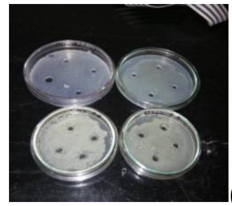

(A)

(B)

Fig 1: Results of Disc method
(A) Doped TiO_2
(B) Pure TiO_2

Disc method results shown revealed that maximum zone of inhibition was found to be in the range 0.6-0.8 cm with a varying concentrations of doped TiO_2.

2.5 Kirby Bauer test

It is an agar diffusion test used for studying sensitivity of bacteria using antibiotic discs, to test the extent to which bacteria are affected by those antibiotics. The antibacterial effect of TiO_2 NPs were performed for comparing the inhibition on *E.coli* by Sm^{3+} doped TiO_2 and pure TiO_2 NPs. Seeded agar was prepared by using 24H culture of *E.coli*, poured to the sterile petriplates and allowed to set. Using sterile cork borer 5 wells of 10 mm diameter were prepared. The wells were filled with pure TiO_2 NPs of concentration 0.2, 0.4, 0.6, 0.8 and 1.0 mg/ml and doped TiO_2 nanoparticles of different mole percentage (1%, 3%, 5%, 7%, 9%) of concentration 0.2, 0.4, 0.6, 0.8 and 1.0 mg/ml. Also, two plates with streptomycin of 0.2, 0.4, 0.6, 0.8 and 1.0 mg/ml served as the control. These plates were allowed to dry and then kept for incubation at 37^0c for 24 hours. The zone of inhibition was observed after 24H.


(A)

(B)

Fig 2: Results of Kirby-Bauer method
(A) Doped TiO_2
(B) Pure TiO_2

The results by Kirby-Bauer test, zone of inhibition in undoped and Sm^{3+} doped TiO_2 was found to be in the range 0.5-1.6cm with the concentration of 1 mg/ml and Streptomycin taken as control.

2.6 Colony forming unit [CFU]

1ml of *E.coli* was aseptically transferred to the petriplates to which LB agar having varying doped TiO_2 concentrations of 0.2, 0.4, 0.6, 0.8, 1.0 mg/ml with Sm^{3+} of 1%, 3%, 5%, 7%, 9% mole % was added. The petriplate without TiO_2 NPs as control. The five petriplates with different concentrations of 20, 40, 60, 80 and 100 µl/ml of pure TiO_2 nanoparticles of LB agar were poured and the plates were incubated at 37^0c for 24 hours. Growth was observed and colonies were counted. CFU's counted in petriplates with varying concentrations of doped and pure TiO_2.

Conc of TiO_2 in mg/ml	Sm^{3+} doped TiO_2	Pure TiO_2
0.2	250	343
0.4	244	321
0.6	143	297
0.8	95	217
1.0	76	170
Control	283	370


(A)

(B)

Fig 3: Colony forming Units in
(A) Doped TiO_2 (B) Pure TiO_2

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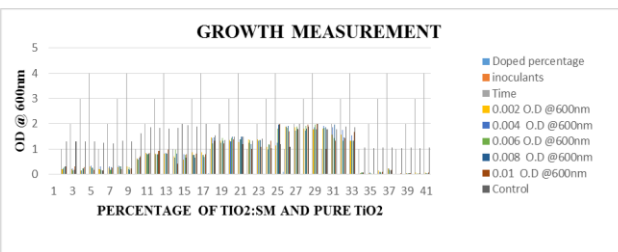
Results indicate that with the rise in concentration of dopant, the growth of colony decreased considerably. The number of colonies formed decides the inhibitory activity of the metal oxide. The more number of colonies indicates low antimicrobial activity. The high number of colonies indicates the high responding antimicrobial activity of the metal oxide.

2.7 Growth curve

Sterile Luria broth was taken in different conical flask 100 µl of duration 24H culture of *E.coli* was inoculated. 20, 40, 60, 80, 100 mg/ml concentrations of Sm^{3+} doped with TiO_2 was added. The flask without TiO_2 serves as control. Flasks were incubated in a shaker incubator at 37ºC and Optical density at 600 nm taken every hour for about 6H of duration.



2.8 Effect of TiO_2 in Liquid Media

The concentration namely 20, 40, 60, 80, 100 µg/ml of TiO_2 was added to prepared and autoclaved 60 ml of Luria broth. 100µl of *E.coli* was aseptically transferred. The flasks were incubated in a shaker incubator at 37^0 C for 18 hours. One flask without TiO_2 served as control. The optical density is noted for every hour for 6H.


(A)

(B)

Fig 4: Results of growth measurement
(A) Doped TiO_2
(B)Pure TiO_2

Fig 4 indicates the growth which was directly proportional to the turbidity of broth medium. It was found that the doped TiO_2 nanoparticles showed decreased turbidity with increasing concentration and hence more bactericidal activity.

Fig. 5 depicts that there was a consistent decrease in the bacterial growth rate in the doped TiO_2 in comparison to the pure TiO_2.

2.9 PXRD Analysis

X-ray diffraction is a non-destructive technique used for the qualitative and quantitative analysis of crystalline compounds. It provides information on phase identification, unit cell dimensions, crystal structure and other structural parameters, such as crystallite size, crystallinity, and phase composition and so on. Nano materials exhibit strong inhibiting effect towards a broadened spectrum of bacterial strains. The inhibitory activity of TiO_2 is due to the photocatalytic generation of the strong oxidizing power when illuminated with UV light at wavelength of less than 385nm for 30mins. TiO_2 particles catalyze the –cidal action of bacteria on illuminator in UV light.

Generation of active free hydroxyl radicals by photo exited TiO_2 particles is responsible for the antibacterial activity. Doped TiO_2 nanoparticles are more inhibitory when compared to pure TiO_2 NPs. The process of doping increases the activity, since the empty sites are filled with Sm^{3+} ions.

Results, Graphical presentation and Discussion
XRD analysis

Fig 6 shows the PXRD peaks which indicate crystallinity, phase and purity. The JCPDS card numbers are verified. The pure TiO2 exists in the rutile phase and with addition of Sm3+, it changes to anatase phase. The particle size was calculated from Debye Scherrer formula and was found to be about 50nm.

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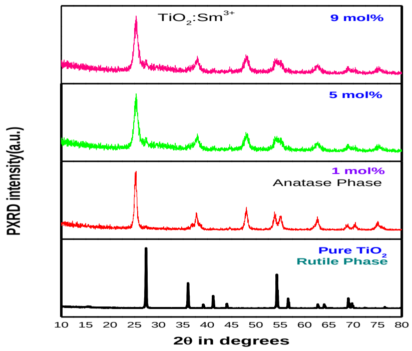

Fig 6: PXRD $_2$ pure and Sm^{3+} doped TiO_2

CONCLUSION

The Comparative study of Sm^{3+} doped TiO_2 NPs by different methods have shown to have bactericidal activity against the bacteria. The effectiveness of these nanoparticles can be enhanced by combining it with the relevant antibiotics minimizing the antibiotic resistance amongst the bacteria. Thus, providing a better scope in future days for combating and treating various diseases.

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