iJRASET
International Journal For Research in Applied Science and Engineering Technology

INTERNATIONAL JOURNAL FOR RESEARCH

IN APPLIED SCIENCE & ENGINEERING TECHNOLOGY

Volume: 5 **Issue:** X **Month of publication:** October 2017

DOI: http://doi.org/10.22214/ijraset.2017.10287

International Journal for Research in Applied Science & Engineering Technology (IJRASET)
ISSN: 2321-9653; IC Value: 45.98; SJ Impact Factor:6.887
Volume 5 Issue X, October 2017- Available at www.ijraset.com

Antibacterial Activity Studies On Mg Doped Ceo$_2$ Quantum Dots

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Abstract: The pure and Mg doped CeO_2quantum dots were synthesized by sol-gel technique and their properties were investigated using XRD, FTIR and HRTEM studies. The structure and size of crystallites were determined using XRD studies. The functional groups of the quantum dotswere analyzed through FTIR spectra. The selected area electron diffraction (SAED) pattern confirms the crystalline nature and cubic structure of the Mg doped CeO_2 quantum dots. Finally, the antibacterial activity of the obtained Mg:CeO_2 quantum dots was also evaluated against Gram positive bacteria staphylococcus aureus (+), Bacillus subtilis (+) and Gram negative bacteria Escherichia coli (-), Pseudomonas aeruginosa (-) using the paper disk diffusion method.

Keywords: CeO_2, Sol-gel, crystalline size, quantum dotand FTIR.

I. INTRODUCTION

Now a days, the synthesis of nanoparticles has found much attention because of increased surface area to volume ratio, modified structure and increased activity compared to micro and macro particles. The application of nanotechnology into biology has opened up immense opportunities in many areas, including tissue engineering, drug delivery, diagnostics, imaging, and fighting bacterial infections.

With the emerging need for novel antimicrobial agents, nanoparticles have been proposed to treat infections as they utilize different mechanisms for killing bacteria than conventional antibiotics, making them promising candidates to overcome current issues facing with antibiotic drug resistant bacteria [1].The nanoparticles of many different elements (such as zinc, copper, titanium, selenium, magnesium, iron oxide and silver) have been studied for their antimicrobial properties [2].

There are many biological characteristics exist in addition to chemical and physical characteristics. The biological pollutants are living pathogenic microorganisms that exist in wastewater, hospital acquired infections etc,. The main microorganisms are bacteria, viruses and protozoa that can cause acute and chronic health effects. Bacteria are single called organisms classified as prokaryotic organisms [3]. Bacillus subtilis, Escherichia coli, Pseudomonds aeruginosa and Staphylococcus areus are the different shaped of bacteria [4]. The size of bacteria can vary according to kind and shape but in general the size ranged from 0.1 to 2 µm [1]. Different diseases such as cholera, typhoid and shigella are caused due to the different kinds of bacteria.

Metal oxides of cerium are basically n-type semiconductorswith cubic crystal structure. Literature often attributes the catalytic activity of cerium oxide, due to its high oxygen storage capacity, high thermal stability, good conductivity and other good electrochemical performances, which is largely due to the multivalence nature of cerium [5]. It is used in wide range of applications such as, catalyst, sensor, solid oxide fuel cells, sun screen cosmetics, bioimaging, biotransformation and antibacterial activity [6-8]. Hence, here an attempt has been made to kill the bacteria using Mg:CeO_2 quantum dots and the zone of inhibitions is measured for various human pathogens.

Generally, CeO_2nanoparticles were synthesized by physical and chemical methods such as hydrothermal, flame spray pyrolysis, sono chemical, microwave, sol-gel, co-precipitation and so on [9-11].In the present study, the sol gel method was used to prepare the pure and Mg doped CeO_2 quantum dots. The as prepared samples were characterized using FTIR, XRD and HRTEM. It was also evaluated the antibacterial activity on Bacillus subtilis, pseudomonas aeruginosa , Staphylococcus aureus and Escherichia coli.

II. EXPERIMENTAL TECHNIQUES

A. Materials

Cerium (IV) nitrate $(NH_4)_2[Ce(NO_3)_6]$ from Nice Chemical company, Ammonium hydroxide (NH_3) obtained from spectrum reagents and chemicals Pvt. Ltd, Citric acid anhydrous ($C_6H_8O_7$) obtained from s–d Fine Chem. Ltd, and deionized water (DI) were used to synthesize pure andMg^{2+} doped CeO_2quantum dots. The purity of the chemicals were above 99%. For all dilution and sample preparation ultrapure water was used. In all the experimental work, acid washed glass wares were used.

B. Methods

The Mg^{2+} doped CeO_2 quantum dots were synthesized using sol-gel method. In a typical procedure, 5.2g (0.4M) of Cerium (IV) nitrate $(NH_4)_2[Ce(NO_3)_6]$ in 25ml of deionized water aqueous mixed with Magnesium acetate (0.1, 0.3, 0.5 and 0.7 M.%) in 25ml of deionized water under constant stirring. The saturated solution of citric acid was added drop wise in to the mixture. After these, NH_3 was added to the precursor solution in order to maintain the pH of the solution. The clear solution was completely turned to a gel after continuous stirring for 4 hours at 70 $^{\circ}$C. Then, the gel was dried and ground into powder. The product was calcined at different temperatures from 200-700 $^{\circ}$C in steps of 100 $^{\circ}$C using muffle furnace. However, the samples calcined at 400 $^{\circ}$C shows higher crystalline then others.

C. Antibacterial activity

The antibacterial activity of Mg doped CeO_2quantum dots were tested against gram-positive (Staphylococcus saprophyticus and Bacillus subtilis) and gram-negative (E. coli and Pseudomonas aeruginosa) bacterias by Kirby-Bauer disc diffusion method. The bacterial suspension was swabbed on the Muller Hinton Agar (MHA) plates using sterile cotton swabs. Sterile wells were prepared with the help of a sterilized stainless steel sterile cork borer. The 100 μL of biosynthesized Mg:CeO_2 nanoparticles were added to the wells at aseptic conditions. The test plates were incubated at 37 ◦C for 24 h. After the incubation period, the zone of inhibition (in mm diameter) was observed and tabulated.

D. Characterization of sol-gel derived CeO_2 nanoparticles

The as prepared pure and Mg doped ceria were characterized using XRD technique. Using X pert PRO diffractometer with a Cu Kα radiation (Kα = 1.5406 Å), the X-ray diffraction (XRD) patterns of the powdered samples were recorded. The morphology and structural details were studied using make: FEI, model: tecnai G2, F30 HRTEM. The FT-IR spectra were recorded in transmission mode by diluting the milled powders in KBr by a SHIMADZU-8400 Fourier-transform infrared spectrometer. The wavelength between 4000 and 400 cm^{-1} was used to assess the presence of functional groups in pure and Mg doped CeO_2 quantum dots.

III. RESULTS AND DISCUSSION

A. X-ray diffraction study

The as synthesised pure and Mg doped ceria were subjected into XRD analysis. The XRD patterns were recorded and are shown in Figure 1. The appeared planes dictated that the ceria has cubic structure with reference to JCPDS file: 34-0394. As seen from the figures, increase in doping concentration increases the FWHM of the planes which means that the size of the crystallite size decreases. The crystallite size were calculated using scherrer's equation [1]

The average nanocrystalline size was caqlculated using Debye-Scherrer's formula.

$$D_{XRD=} \frac{K\lambda}{\beta cos\theta} \qquad (1)$$

Where D is crystallite size, β is full width of half maximum (FWHM), K is factor (K=0.9 in this work), λ is the wavelength of incident X-rays (λ = 0.15406nm)[12].

The crystallite sizes of the pure and various concentration (0.1, 0.3, 0.5 and 0.7 M.%) of doped (Mg) ceria quantum dots are 10.98, 6.94, 16.66 13.89 and 10.42 nm respectively. Since the crystallite size of the particles are less compared to twice the value of the exiton Bohr radius of the ceria (7-8 nm), the particles may be called quantum dots. Thus, from the above result, it is observed that the sample having 0.1 M.% of Mg doped ceria has less crystallite than pure and other doped samples. The detailed results were published in our earlier publication [13]

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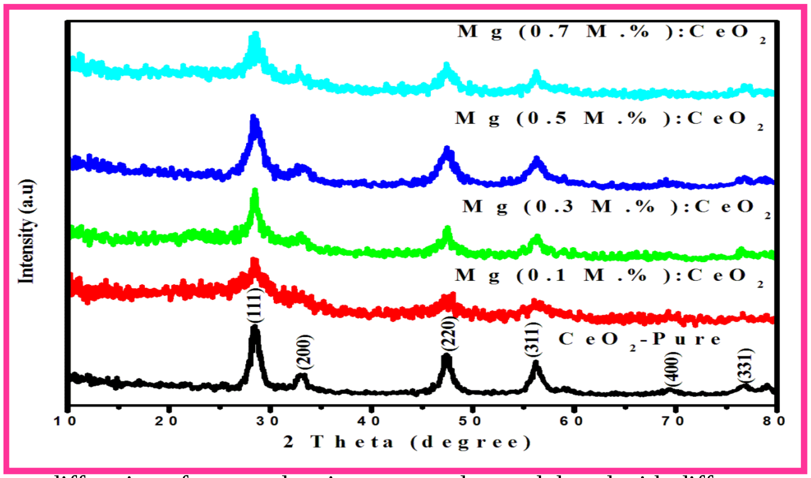


Fig. 1X-ray diffraction of pure and ceria quantum dots and doped with different concentrations of Mg

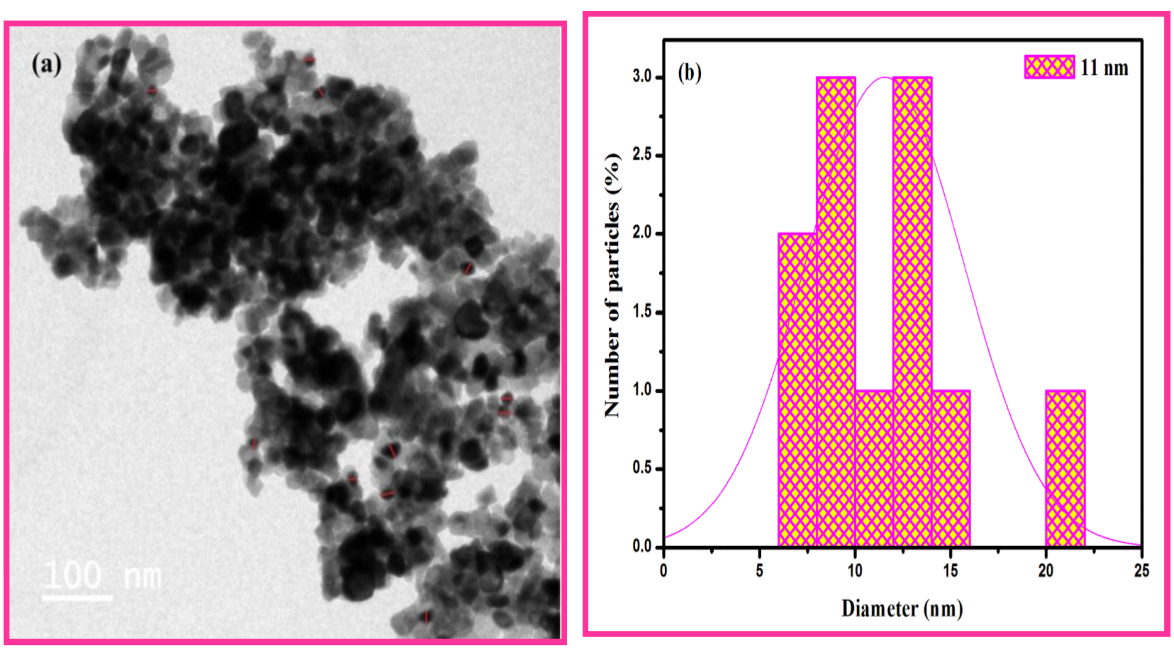
B. High resolution transmission electron microscopy (HR-TEM)

Among the pure and various concentration of Mg doped ceria quantum dots, 0.1 M.% of Mg doped ceria quantum dots are having less crystallite size. Hence, this sample is taken for HRTEM analysis.

The HRTEM image provides the detail morphology of Mg (0.1 M.%) doped CeO_2 quantum dots, which are shown in Fig. 2(a). Fig. 2(b) shows the well dispersed quantumdots with a size distribution ranging between 5 to 25 nm and the maximum number of particles range are in 11nm. Fig. 2(c) shows the lattice fringes and overlapping of two crystals. Two adjacent fringes possess the average fringe distance of 17.49 nm. The selected area electron diffraction (SAED) pattern confirms the crystalline nature and cubic structure of the Mg doped CeO_2 quantum dots shown in (Fig. 2 (d)).The results are will matched with XRD observation.

C. Fourier transform infrared spectroscopy (FT-IR)

Fourier transform infrared spectroscopy (FT-IR) was usually employed as an additional probe to evidence the presence of OH groups as well as other organic and inorganic species. The pure and Mg (0.1, 0.3, 0.5 and 0.7 M.%) doped CeO_2quantum dots materials were characterized by the FTIR spectroscopic techniques in the range 4000 - 400 cm^{-1} which identify the chemical bonds as well as functional groups in the compound. For the as-prepared samples in Fig. 3, the large broad absorption bands positioned at 3438 cm^{-1} and 1556-1383 cm^{-1} are associated to the symmetric stretching (νOH) and bending modes of (δ OH) internally bonded



water molecules, respectively [14]. The band at 2344 $cm^{-1}$are due to the stretching vibration of the C–H bond of organic compounds [15]. The FTIR absorption bands at about 863 cm^{-1} are similar to those of commercial CeO_2quantum dots. The band seen at 456 cm^{-1} Mg doped spectra show the existence of vibration of Mg^{2+} ions in the network vacancies [16, 17].

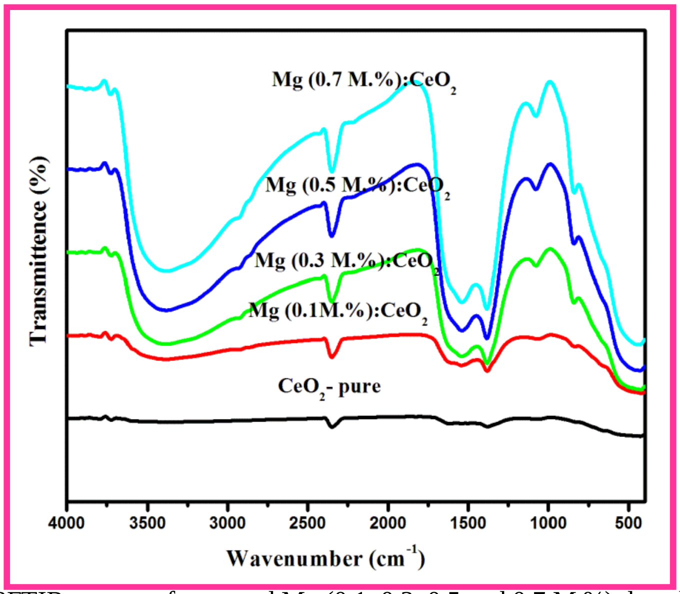
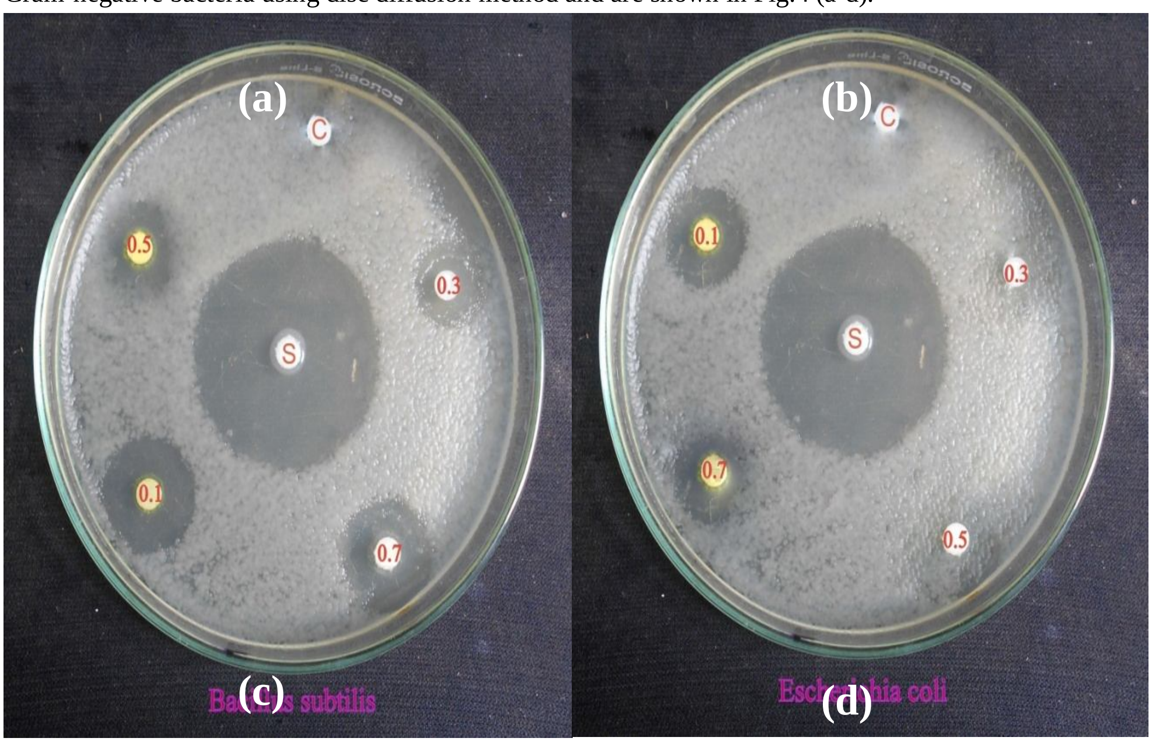


Fig.3FTIR spectra of pure and Mg (0.1, 0.3, 0.5 and 0.7 M.%) doped CeO_2 quantum dots.

D. Antibacterial activity

In the present study, the antibacterial effect of prepared Mg doped ceria quantum dots was found on different types of bacteria such as Bacillus subtilis, Staphylococcus aureus (Gram-positive), Escherichia coli and Pseudomonas aeruginosa (Gram-negative).

The bacteria inhibition zone for Mg (0.1, 0.3, 0.5 and 0.7 M.%) doped CeO_2 quantum dots were examined against both Gram-positive and Gram-negative bacteria using disc diffusion method and are shown in Fig.4 (a-d).



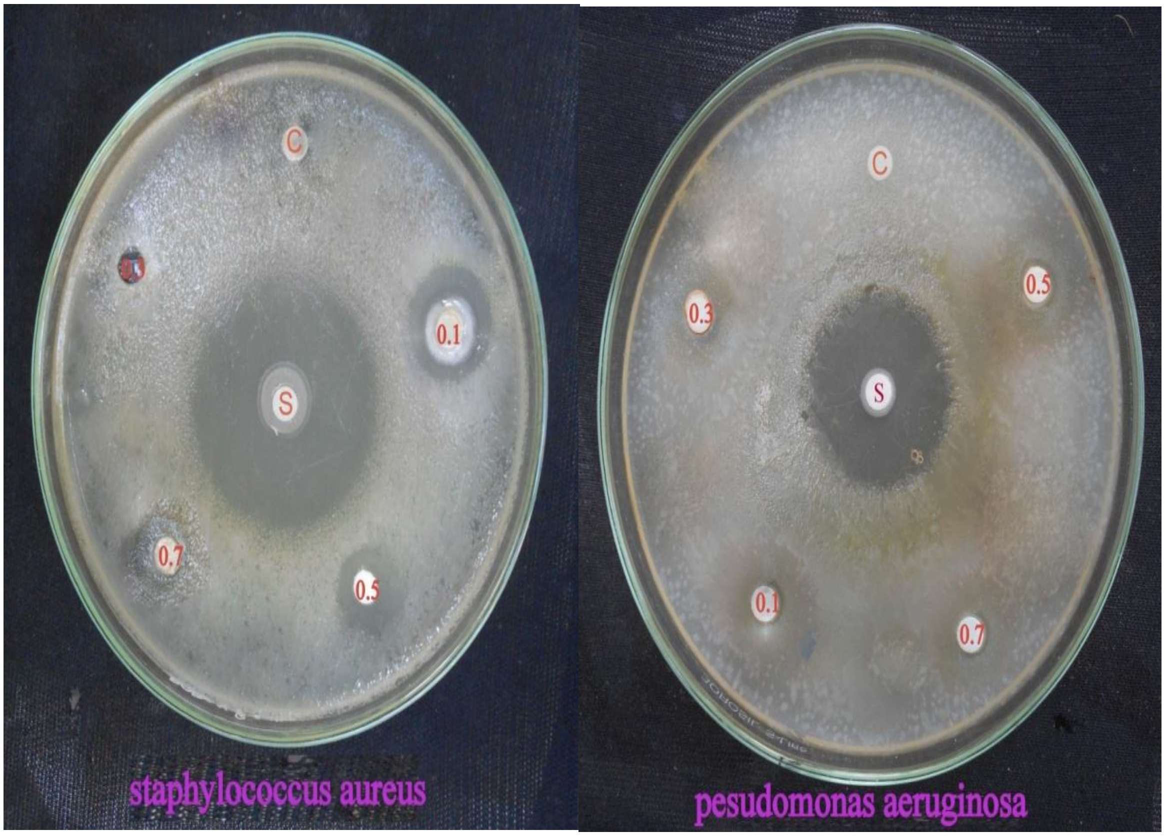


Fig.4. antibacterial activity of (a) Bacillus subtilis, (b) Eschericha coli, (c) staphylococcus aureus and (d) Pseudomonas aeruginosa of Mg (0.1, 0.3, 0.5and 0.7 M.%) doped CeO_2 quantum dots: Control (C), Standard (S).

The diameter of the inhibition zones are 18, 13, 14 and 11mm, 10, 04, 03 and 02 mm, 11, 08, 07 and 05 and 15, 09, 11 and 08 mm against B. Subtilis, E. coli, S. aureus and P. aeruginosa respectively. The results are summarized and presented in Table 1. It is observed that dimethyl sulfoxide(sample of C in Fig. 4 (a-d)), which was used as a control matrix, exhibited no antibacterial activity when compared with Mg (0.1, 0.3, 0.5and 0.7 M.%) doped CeO_2 quantum dots. According to Fig. 4 (a-d), the Mg (0.1 M.%):CeO_2 quantum dots showed better antibacterial activity against B. Subtilis, E. coli, P. aeruginosa and S. aureus, which may be due to its small crystallite size. However, the antibacterial activity in Gram positive is higher than Gram negative bacteria.

This is because, the Gram-positive bacterial cell wall contains a thick layer of peptidoglycan, which is attached to teichoic acids, and this may be the reason for the interaction with CeO_2 quantum dots in antibacterial activity. The zone of inhibition effect of antibacterial activity depends upon the concentration of Mg:CeO_2 quantum dots. The observed results could be attributed to a binding of metal and metal oxide nanoparticles on to the bacterial cell wall due to the electrostatic attraction between the negatively charged bacteria and the positively charged nanoparticles. This interaction not only inhibits the bacterial growth, but it also induces the generation of the reactive oxygen species (ROS), which leads to cell death. At low concentration of nanoparticles, the interaction of particles with the cell wall of bacteria decreases and at the high concentrations of the particles, the aggregation probability of particles increases, as a result, the effective surface to volume ratio of particles and so the resulting interaction between particles and the cell wall of bacteria decrease [18].

The effect of Mg quantum dots on the bacterial cell are complicated. However, there are various mechanisms on the action of Mg quantum dots on the bacterial cell. Some of these mechanisms were summarized and presented as follows: (i) the ability of Mg quantum dots to anchor to the bacterial cell wall and then penetrate it [19], (ii) the formation of free radicals by the Mg quantum dots which can damage the cell membrane and make it porous [20] ,(iii) releasing the Mg ions by the nanoparticles which can interact with the thiol groups of many vital enzymes and inactivate them [21], and (iv) the nanoparticles can modulate the signal transduction in bacteria which stops the growth of bacteria [22].

From the table 1, it is also observed that the variation of inhibition zone depends upon the particle size of the Mg:CeO_2 quantum dots. As particle size decreases, the diameter of the inhibition zone increases and vice versa.

Table 1 activity [disc diffusion method] of Mg doped CeO_2 quantum dots

S. No.	Bacteria	Zone of inhibition (mm)					Crystallite size (D)
		Bacillus subtilis(+)	Escherichia coli(-)	Staphylococcus aureus (+)	Pseudomonas aeruginosa(-)	Control (DMSO)	
1	Standard Antibiotic Disk	25	25	25	22	-	
2	Mg(0.1 M.%):CeO_2	18	13	14	11	-	6.94
3	Mg(0.3 M.%):CeO_2	10	04	03	02	-	16.66
4	Mg(0.5 M.%):CeO_2	11	08	07	05	-	13.89
5	Mg(0.7 M.%):CeO_2	15	9	11	08	-	10.42

IV. CONCLUSIONS

The pure and Mg doped ceria quantum dots are successfully synthesized by sol - gelmethod. The crystallite sizes of the pure and various concentration (0.1, 0.3, 0.5 and 0.7 M.%) of doped (Mg) ceria quantum dots are 10.98, 6.94, 16.66 13.89 and 10.42 nm respectively. Since the crystallite size of the particles are less compared to twice the value of the exiton Bohr radius of the ceria (7-8 nm), the particles may be called quantum dots. The selected area electron diffraction (SAED) pattern confirms the crystalline nature and cubic structure of the Mg doped CeO_2 quantum dots.The results are will matched with XRD observation.The FTIR absorption bands at about 858 cm^{-1} are similar to those of commercial CeO_2quantum dots. Thus it is concluded that the Mg:CeO_2 quantum dots showed better antibacterial activity against B. Subtilis, E. coli, P. aeruginosa and S. aureus, which may be due to its small crystallite size.

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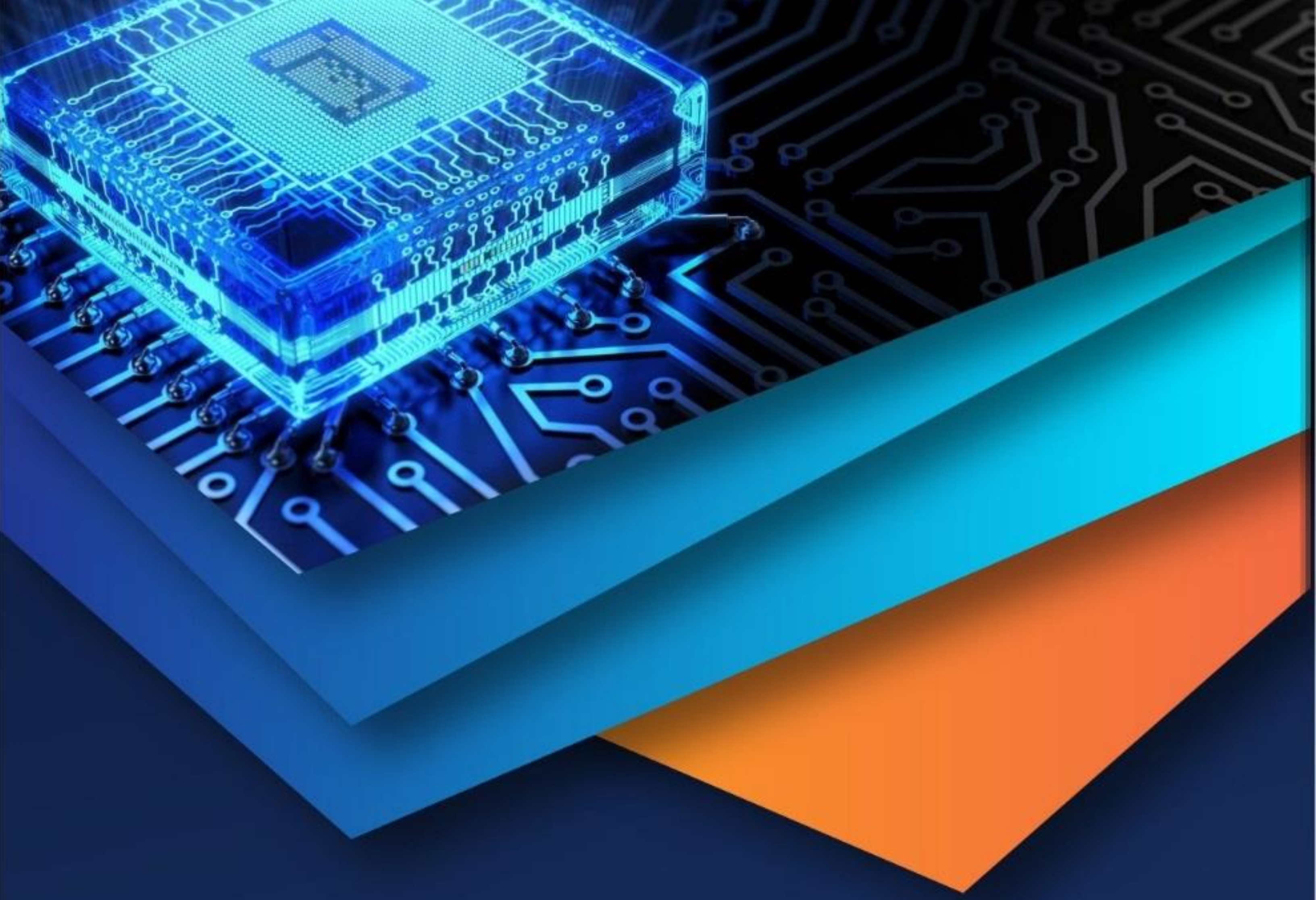

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