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Synthesis, characterization and degradation of Boron, Cerium and silver ternary doped titanium dioxide photocatalyst via EDTA citrate method using ampicillin antibiotic under Sunlight

Yash Mishra, Dr Hari Mahalingam
National Institute of technology Karnataka,Surathkal
(Department of Chemical Engineering)
yash250025@gmail.com

Abstract

Nowadays, we can see that in river water, traces of antibiotics can be found, which is an emerging problem. Also, pharmaceutical companies' wastewater contains antibiotic traces present in it in a significant amount which makes it an excellent experimental domain to work upon. Which is very harmful if taken by humans without its treatment, so to treat it as early as possible is very necessary, else the bacteria emerging in that water will be converted to superbugs and then curing the disease from that bacteria will be exceedingly difficult as they have resistive power to that antibiotic. For that, we have prepared a tri-doped photocatalyst by doping boron cerium and silver in a titanium dioxide structure. It can work under sunlight light because the presence of silver in this boron amount is increased so that it can satisfactorily degrade antibiotics. Cerium is for water disinfection in the further catalyst. Its amount was also increased. Then the characterization analysis was performed with the help of DLS analysis with the help of a nanoparticle size analyzer, and we got particle size in the range of 115 to 600 nanometer XRD analysis. We got a band gap Of 2.3 to 2.4 electron Volt.BET surface area analysis showed us a surface area of about 25 m2/g.So instead of the UVA lights now, it was performed under the sunlight and the degradation percent was increased significantly to approx 70 percent.

Keywords :-PhotoCatalyst, antibiotic, Ampicillin,Sunlight,EDTA Citrate Method,Ternary –doping

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INTRODUCTION

Rapid modernization were the most important contributors to water pollutants for the duration of the world. nowadays, pharmaceutical residues detected in the μg/L-ng/L variety in surface water,groundwater, and consuming water resources are considered as emerging contaminants due to the extended opportunity of improvement of superbugs. A recent newspaper article has pointed out the presence of superbugs in the river Ganga, a primary river in India. some other article has highlighted the pollution due to antibiotics in rivers everywhere in the international, with the concentrations often exceeding the threshold limits. nearly 90% of the antibiotics are excreted in their determined form from the human body, which subsequently reaches the sewage treatment plants . The considerable assets of antibiotic residues because of the anthropogenic activities are partially treated or untreated effluents from sewage treatment flowers, hospitals, and pharmaceutical manufacturing industries.Fluoroquinolones, carbapenems, and cephalosporins are the maximum generally said class of antibiotic residues in the effluent treatment vegetation of India. Ampicillin belongs to the fluoroquinolone magnificence of antibiotics, and they're used to treat a variety of bacterial infections. Their presence in water bodies has been suggested in the range of 5,000–31,000 μg/L and 251,000 μg/L, respectively . The predicted No-impact concentration-minimal Inhibitory concentration (PNEC-MIC) for the prevention of growing resistance is postulated based on the technique endorsed by Bengtsson-Palme and Larsson (2016) and by way of the Antimicrobial Resistance (AMR) Industry Alliance (2018). The PNEC for ampicillin is 0.4 μg/L . The capacity adverse results of those antibiotic residues are vegetation irrigated with contaminated water ends in bio-accumulation within the food chain (from the farm to the table and then into the human body), the aquatic existence is affected due to quandary of the mild penetration, sewage drains get clogged with the bacterial biofilms due to the accumulation/sorption of antibiotic residues onto the sewage sludge, and the switch of pathogenic (antibiotic-resistant) genes into wholesome microbes promote an growth in superbugs . therefore, there is a pressing want to deal with these wastewaters. diverse physical, chemical, and organic remedy methods like membrane strategies, reverse osmosis; activated carbon, chlorination; cardio, and anaerobic strategies had been in existence for the remedy of pharmaceutical wastewaters. The fundamental drawbacks of these remedy methods are clogging of membranes, carbon regeneration, obstacle of size exclusion range, disposal problems (physio-chemical remedy), biomass accumulation, sluggish technique, and requirement of long startup intervals (biological treatment). due to the poor degradability of antibiotics and the unsuitableness of wastewater remedy plants to deal with that pollution, there's

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a need for the improvement of opportunity sustainable remedy methods . Photocatalysis, with the potential to degrade the organic contaminants in water and wastewater, serves this cause . Semiconductor-based totally photocatalysis is one of the superior oxidation processes (AOPs) for the removal of organic contaminants via the generation of powerful oxidizing marketers [14]. TiO2 is the maximum broadly used photocatalyst as it's far effectively available, non-poisonous, chemically, and mechanically solid. but seen light usage because of the huge bandgap of 3.2 eV is a prime quandary. the usage of seen mild active photocatalysts is gaining importance in recent times, which may be executed through engineering the bandgap of semiconductors through doping. diverse metals (transition-Fe, Cu, Co, Zn, Mn, Mo, Ni, W; rare earth-Ce, Gd, European, Y; and others-Li, Ge, Si, Sn, Pb,) or non-metals (B, C, N,S, P) help in narrowing the bandgap for effective usage of visible light, and gives splendid chemical stability .it's miles glaring that the presence of rising pollution mainly antibiotic residues in water bodies and the ensuing linkage to the increasing antimicrobial resistance is of great challenge and on this context, it'd be suited to have a seen mild photocatalyst that is simultaneously effective for each photo-degradation in addition to disinfection. preserving this goal in mind, the dopants are narrowed down to cerium (Ce) and boron (B) for the reasons said below. uncommon-earth metallic doping like cerium results in the narrowing of the bandgap and promotes the adsorption capacity for this reason, enhancing photocatalytic hobby by way of providing better touch between pollutant and photocatalyst. Ce-TiO2 photocatalysts had been suggested for the degradation of phenol, chlorophenol, polyvinylpyrrolidone, and methylene blue and disinfection of Staphylococcus aureus. some of the various non-metal dopants, B ions occupy both the substitutional and interstitial lattice positions of TiO2, the former position facilitates in narrowing of the bandgap, introduction of new power ranges and transferring of the absorption part closer to visible light even as the latter role promotes effective electron-hollow separation and reduces the recombination rate. it's also a powerful disinfectant . B-TiO2 photocatalysts were suggested for the degradation of p-nitrophenol, Orange II azo dye, and disinfection of Staphylococcus aureus and Escherichia coli. other than those, photocatalytic degradation of antibiotics (ampicillin) and dyes (Rhodamine B, RhB) had been suggested via [25–27] the use of l. a./Cu/Ze trimetallic nano photocatalyst BiOBr/BiFeO3, and Sr/Ce/AC bimetallic nanocomposite respectively. numerous strategies for the synthesis of doped photocatalysts like sol-gel, hydrolysis, hydrothermal, solvothermal, and co-precipitation have been pronounced. The EDTA-citrate technique affords a higher catalyst yield and additionally improves the properties of the catalyst debris. EDTA forms a hoop structure with a maximum of the metallic ions and enables the molecular level of mixing. there's little or no or no literature on the synthesis of Ce-TiO2 ,Ag-TiO2 and B-TiO2 using this method. The photocatalytic degradation of ampicillin by means of TiO2 was studied , and a few researchers have mentioned the usage of doped TiO2, BiOBr and Fe debris . A summary of the research carried out on

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the photocatalytic degradation of ampicillin is provided in tables S9 - S10 of supplementary. in this manuscript, the photocatalytic degradation of ampicillin under sunlight with the use of a series of B,Ag and Ce-doped TiO2 photocatalysts synthesized by the EDTA-citrate approach has been finished at the side of the photocatalytic disinfection of E.coli. The precise stress MTCC 9541 used for the disinfection examination is remoted/derived from the river Ganga in view of the full-size antimicrobial resistance present in this river .

Materials

The following chemicals were used without further purification in the synthesis of the catalysts TiO2 (Degussa P25, 99.9% pure) from Evonik (Japan), Boric acid, EDA, citric acid and ammonia solution from Loba Chemie Pvt Ltd. (India), Cerium nitrate and silver nitrate from Sigma-Aldrich (India), Ampicillin from Sigma-Aldrich (USA). Distilled water was employed in the preparation of all solutions.

Synthesis of Nanoparticles

Two ternary doped TiO_2 catalysts were made using the modified sol-gel method employing EDTA & citric acid as the chelating and complexing agents respectively. A mole ratio of 1:1:1.5 being the ratio of metal ions to EDTA and citric acid was used in the synthesis. EDTA $C_{10}H_{16}N_2O_8$) solution was made using water and ammonia. Stoichiometric quantities of boric acid (H_3BO_3), cerium nitrate ($Ce(NO_3)_3{\cdot}6H_2O$), silver nitrate ($AgNO_3$), and titanium dioxide (TiO_2) in aqueous form was added to the solution & stirred. Then, solid citric acid ($C_6H_8O_7$) was added to the above mixture. Ammonia is used for adjusting the pH to 9, and the resulting mixture was agitated gently till an organometallic gel formed. Drying of the gel was done a laboratory oven at 150 °C for 24 h. The dried sample was finely crushed and subjected to calcination in a muffle furnace at 350 °C for 12 h, followed by calcining at 600 °C for 5 h. The powder thus obtained was stored in an air-tight bottle for further use. In this work, two ternary doped catalysts are synthesized and denoted as 2B-0.1Ce-0.06Ag-TiO_2, 2B-1Ce-0.06Ag-TiO_2.Due to the use of water as a solvent thus replacing the volatile organic solvents usually employed, the process can be considered as relatively green . It may be noted that Boron is an excellent disinfectant and cerium promotes the adsorption capability of the catalyst while silver has excellent antimicrobial properties and surface plasmon resonance enabling the catalysts functioning under visible or solar light. We wanted to examine in detail, the effect of increased amounts of cerium and silver in this work keeping the amount of boron dopant is constant.

LEACHING ANALYSIS :-

Leaching analysis were performed on both photo-catalyst to deduce the amount of dopants and titanium being leached while performing

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analysis.Results obtained are shown below

BORON RESULTS
2 B 1 CE 0.06 AG 0.54 PPM
2 B 0.1 CE 0.06 AG 0.29PPM

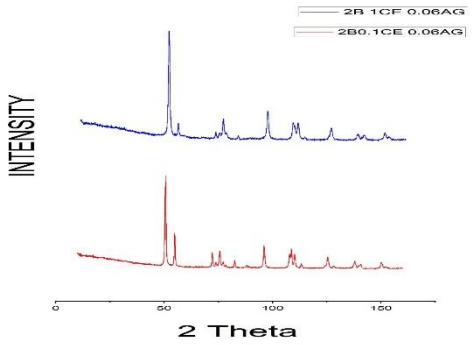
CERIUM RESULTS
2 B 1 CE 0.06 AG 0.04 PPM
2 B 0.1 CE 0.06 AG 0.00 PPm

TITANIUM RESULTS
2B 1 CE 0.06 AG 0.24 PPM
2 B 0.1 CE 0.06 AG 0.11 PPM

SILVER RESULTS
2B 1 CE 0.06 AG 0.00 PPM
2 B 0.1 CE 0.06 AG 0.00 PPM

XRD ANALYSIS :-

The XRD spectra of two doped photocatalysts are shown in below fig . As can be seen, the two spectra are nearly identical, confirming the correctness of the synthesis procedure. The peaks observed at □ = 24.5 and 47° indicate the anatase phase, while the slight rise immediately following this at about 29° suggests the presence of the cerium oxide (111) phase. Since silver ion is relatively large compared to titanium and cerium, besides being present in meagre amounts, the XRD spectra do not show the presence of silver. Boron is also not indicated.



DRS ANALYSIS :-

Diffuse reflectance spectroscopy analysis was performed on the synthesized catalysts to obtain the band gap energy values as determined from the Tauc plot. In the analysis, TiO_2 is considered as an indirect semiconductor and the calculations are done on this basis. The formula used is

$$\alpha h\nu = A\left(h\nu - E_g\right)^{n/2} \quad (1)$$

where, E_g - band gap energy (eV), α - absorption coefficient, h - Planck's constant, ν - frequency of light, A - constant, n = 4 (for indirect transition). The plot of $(\alpha h\nu)^{1/2}$ versus (hν) energy determines the band gap energy values which is obtained by extrapolation of the linear portion of the curve to the x-axis. Relative to the band gap energy value of TiO_2 (3.4 eV) .

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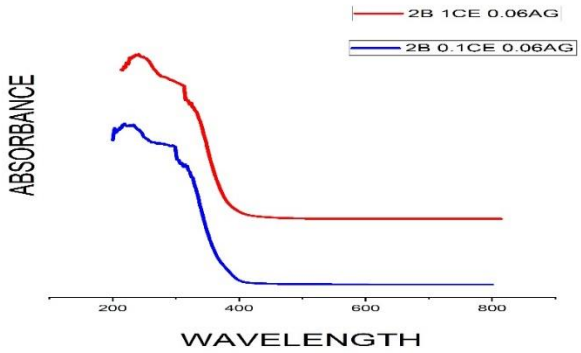
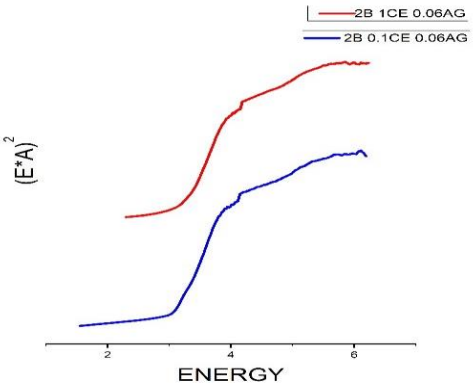


The characterization analysis results obtained from DRS analysis were used for plotting the graph of Absorbance Vs Energy



The characterization analysis results obtained from DRS analysis were used for plotting the graph of (E*A) ^2 vs energy.

CALIBRATION OF AMPICILLIN ANTIBIOTIC

The photocatalytic degradation of ampicillin under sunlight irradiation is used to evaluate the performance of TiO2 and the synthesized photocatalysts. A irradiation time of3 hours with a mean intensity of 200 ± 20 lx and a temperature of 28°C is used in the conduct of experiments. The volume is 200 mL of 10 mg/L, 20 mg/L, and 30 mg/L pollutant concentration (Ampicillin) and the catalyst loading is chosen as 1 g/L. The samples are kept in the dark for 30 minutes to attain complete adsorption before starting the illumination. Liquid samples are taken at regulartime intervals and filtered & centrifuged to remove the solid catalyst particles.

The percentage of Ampicillin was calculated according to the following equation:

Ampicillin degradation = (c0−c/c0× 100%)

where Co – Initial concentration,

C - Final concentration

The calibration analysis was performed for the ampicillin antibiotic with the help of UV Spectro photometer and the respective absorbance vs wavelength graph was plotted as shown in below figure and also absorbance Vs concentration graph was plotted for the

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ampicillin antibiotic as depicted in the second figure

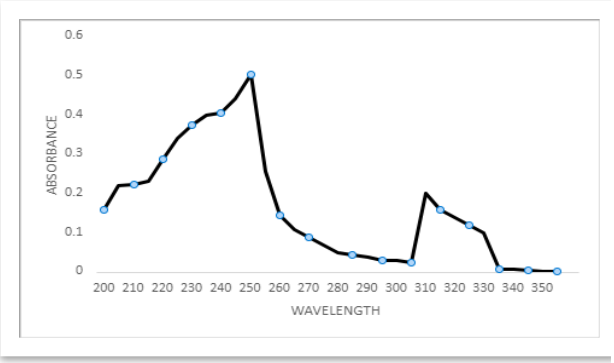


Fig :- Absorbance Vs Wavelength curve for Ampicillin Antibiotic

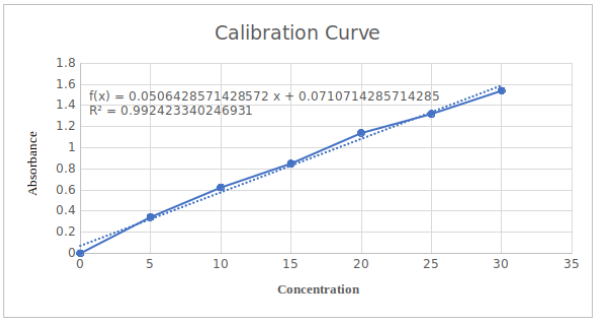


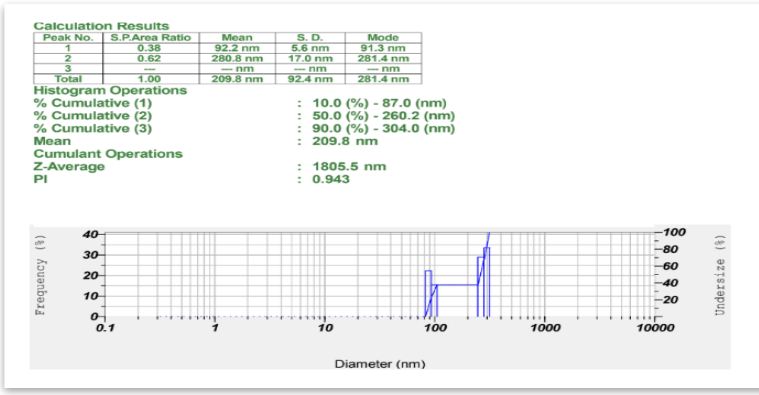
Fig Absorbance Vs Concentration graph for Ampicillin Antibiotic

DLS ANALYSIS :-

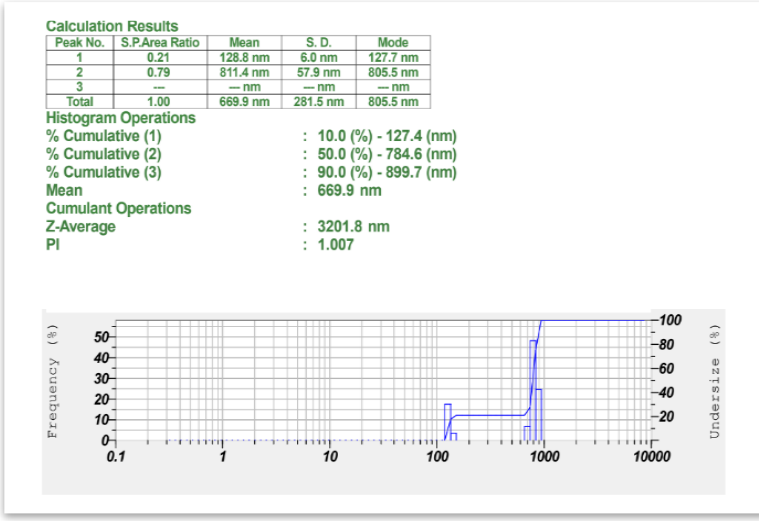
The particle size was more than the previously formed catalyst which is usual as the amount of boron is increased in the catalyst and also in comparison amonf gboth photo-catalyst second one was having more size since it has more amount of cerium content being doped.

As the graph below clearly shows the size of few nano particles is more than the desired value it is because the boron may not have doped correctly in the Ti-O2 or due to the formation of the agglomerates.

ATOMIC CONFRIGUATION :- 2B 0.1B 0.06AG



ATOMIC CONFRIGUATION:-2B 1B 0.06Ag-TiO2



PARTICLE SIZE RESULTS

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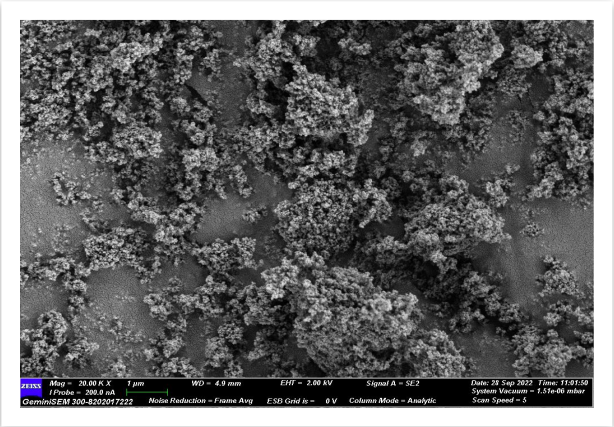
2B 1 CE 0.06 AG	209.8 NM
2 B 0.1 CE 0.06 AG	669.8 NM

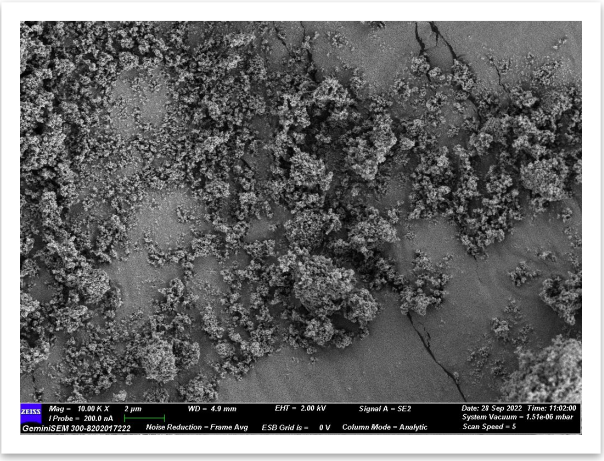
SEM Analysis

The surface morphologies of undoped TiO2, B-Ce-Ag-TiO2, and B-Ce-Ag samples are characterized by SEM and the images are shown below .No specific treatment is provided to the TiO2 sample prior to SEM analysis as it was commercially available. However, in the case of synthesized ternary doped catalysts prior to SEM analysis, they are sonicated for 10 min and then dried. The ternary doped photocatalyst particles appear as loosely packed irregular/elongated aggregates with a coarse surface, whereas TiO2 particles appear as closely packed small aggregates with a smooth surface. In the doped catalysts, a large number of nanoparticles are assembled to form aggregates on the primary particles. Higher agglomeration with an increase in the Ce dopant concentration can be attributed to the larger ionic radius of Ce than that of Ti, implying that it cannot enter the lattice of TiO2 and therefore, Ce peaks are observed in the XRD spectra However, an opposite trend of a decrease in agglomeration (as well as particle size) with an increase in boron dopant concentration was observed. The decrease in agglomeration and particle size can be attributed to the smaller ionic radius of B than that of Ti due SEM Images of the first ternary doped photo catalyst were analysed and best 4 images among them were selected.

SEM Results.

ATOMIC CONFRIGUATION:- 2B 0.1CE 0.06AG





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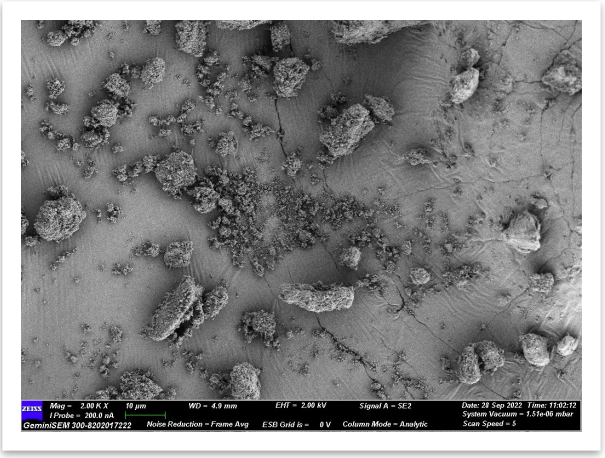

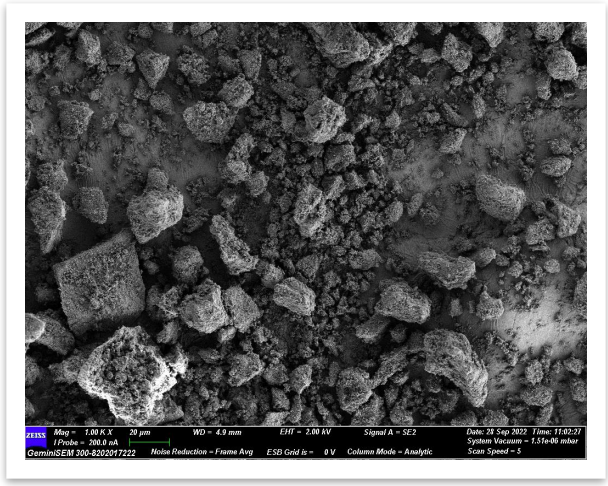

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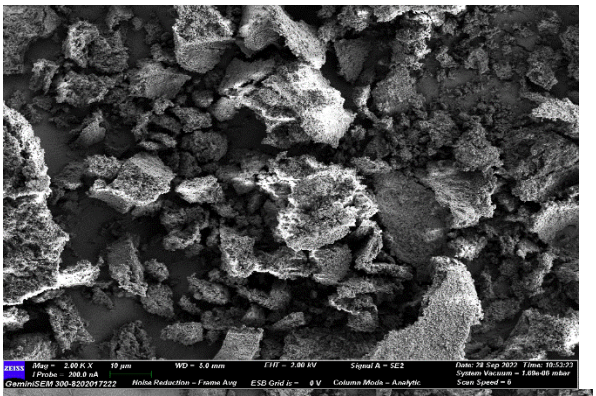

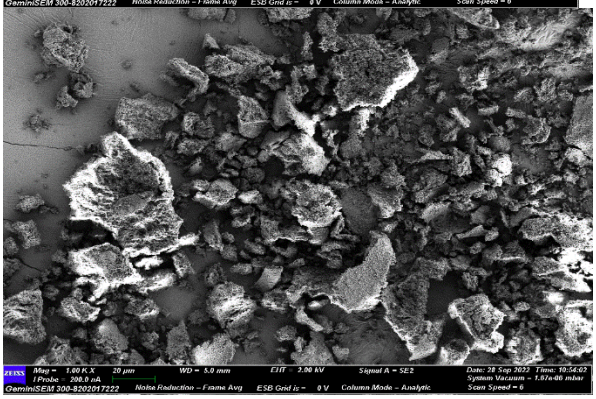

ATOMIC CONFRIGUATION :-2B 1CE 0.06AG

The images of the second photo catalyst were also analyzed and best 4 images were selected from them. Agglomerates can be seen in the below images

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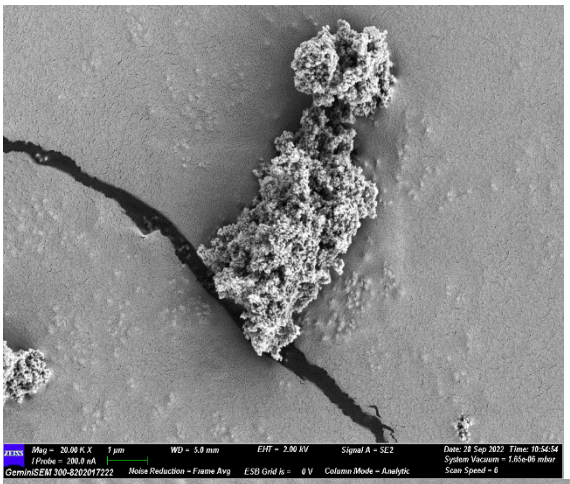
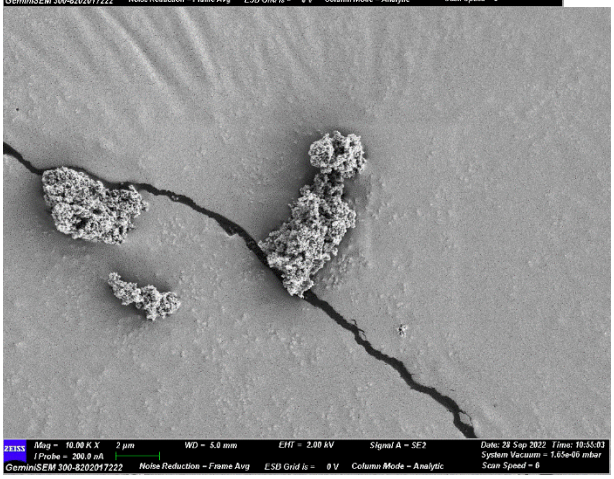
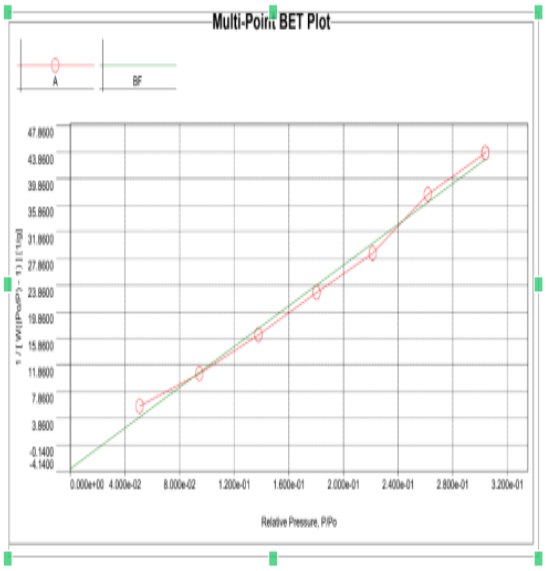






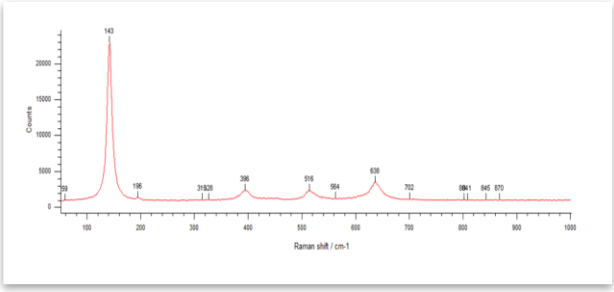
BET SURFACE AREA ANALYSIS: -

Langmuir and BET plot analysis gave the desired results. The pore size and pore volume were also found in the desired range.The outgas time was 6 hrs.The method used for the BET surface analysis is physisorption method. The model of the BET surface area which describes the quantity of adsorbed gas as a function of the relative pressure, p/po.In the BET suface area analysis we use the area per molecule for the calculation olf whole BET surface area.

GOT bet surface area as 23m^2/g.

RAMAN SPECTROSCOPY:-

Raman spectroscopy is employed to understand the crystal structure of the photocatalysts through vibrational modes, and the spectrum is depicted below. The anatase Raman modes in the undoped and doped samples are found at: Eg(1)-143 cm–1, Eg(2)-196 cm–1, Eg(3)-636 cm–1, B1g-396 cm–1, A1g + B1g-5176cm–1. The intensity of anatase Raman mode, especially Eg(1), is found to be higher in all the photocatalysts . It is noted that as in the XRD, here also, the boron peaks are not detectable.



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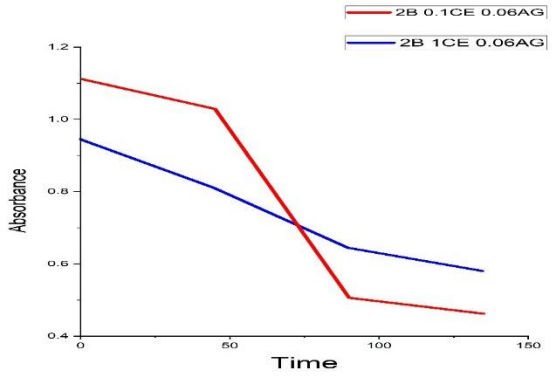
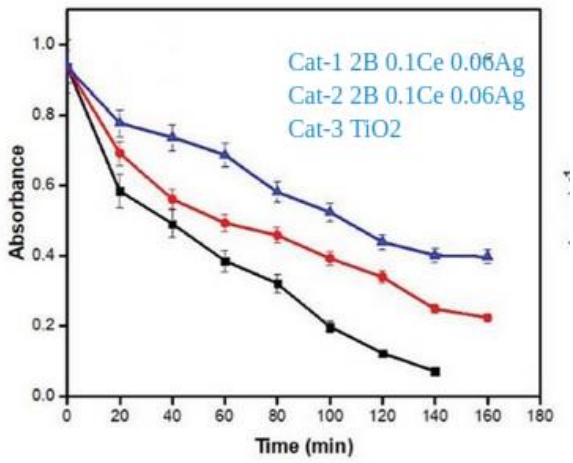
DEGRADATION RESULTS:-

The degradation studies of ampicillin for best-performing ternary-doped photocatalysts, i.e., B2Ce1Ag0.1TiO2 and B2Ce0.1Ag0.06TiO2 were carried out under sunlight at room temperature. The degradation in the presence of TiO2 shows good results, whereas the remaining ternary-doped catalysts are not performed significantly under the sunlight. The results are shown below in the graphs.The graph above shows the degradation of ampicillin under sunlight in which the effect of photocatalytic degradation of ampicillin was studied at 10 ppm, 20 ppm, and 30 ppm using ternary-doped catalysts i.e., B2Ce1Ag0.1TiO2 and B2Ce0.1Ag0.06TiO2. To compare the ternary-doped catalyst performance with TiO2, the degradation study was carried out with TiO2. An optimum value is noticed for the co-doped catalyst from previously done research work, while an increasing trend is observed for the Ternary-doped catalysts. TiO2 showed almost 100% degradation at an optimum catalyst loading of 1g/l in 10ppm, 20ppm, and 30ppm concentrations of ampicillin. After placing the whole system of each concentration for 30min in dark conditions, the degradation was observed in 20% for 30ppm solution and 35% for 10ppm, and after 90min the ampicillin is almost degraded to 100% for all concentrations (10ppm, 20ppm, 30ppm). After 90 min, the degradation of ampicillin by TiO2 was more than 96%. For ternary-doped catalyst 2B-1Ce-0.1Ag-TiO2, after 30min of placing in dark conditions, the degradation was observed at a minimum of 16% at 50ppm and a maximum of 27% at 10ppm. After 120min of placing the whole system under sunlight, the degradation was observed at a minimum of 45% and a maximum of 68%. Among both photo catalyst one with more dopant concentration of cerium was best performing.



CONCLUSION: -

The solar light-driven photocatalytic degradation of ampicillin using B-TiO2, Ag-TiO2 and Ce-TiO2 has been carried out i



n this study. The photocatalysts synthesized by the EDTA-citrate method showed particle size in the nanoscale range and surface area in the field of 25 m2/g. T The XRD diffraction peaks of Ce suggest the presence of Ce on the surface of TiO2

due to its large radius, and the absence of prominent boron peaks indicates the uniform dispersion of B in TiO2. The bandgap values obtained are in the range of 2.4–2.7 eV, which suggests an introduction of a new energy level for effective charge separation and reduction of recombination. The enhancement in the photocatalytic activity of Ce-TiO2 and Ag-TiO2can be ascribed to the higher adsorption ability of these photocatalysts when compared with boron as the water adsorbed surface generates more hydroxyl radicals and acts as a photoexcited hole trap. An enhancement in the photocatalytic activity of B-TiO2 to the presence of B in the interstitial lattice position where there is a formation of a shallow level below the conduction band as confirmed from DRS, which reduces recombination and due to the presence of Ti3+, which acts as trapping sites of photogenerated electrons. The degradation of ampicillin is found to obey pseudo-first-order kinetics with high k values and R2, suggesting an increase in the rate of the reaction. The photocatalytic degradation of ampicillin has occurred mainly through decarboxylation, hydroxylation, defluorination, and transformation of the piperazine ring with an intact quinolone moiety which finally forms lower molecular weight/less harmful products. The absence/decrease in the intensity of the pollutants peak in the HPLC/LC-MS data validates their effective photo catalytic degradation under sunlight. However, the COD reduction in some photocatalysts was found to be lesser than the degradation observed through a spectrophotometer, which might be due to the formation of other, less harmful and stable organic compounds as observed in the LC-MS. The photocatalysts are found to be durable for up to three consecutive recycles. The trapping experiment results implied that the e– and OH are the dominant active species involved during the photocatalytic degradation of ampicillin. From the above analysis, one at. % Ce-TiO2 and one at. % B-TiO2 are found to be the best-performing photocatalysts, which can be due to the higher adsorption rates in cerium and interstitial lattice position of boron, along with a decrease in the bandgap. Their disinfection efficiencies (of the best-performing catalysts of degradation) are found to be in the range of 97–99.99%. Hence, these sunlight-active photocatalysts could be the facile and sustainable solution for the emerging problem of antimicrobial resistance. The immobilization of these photocatalysts onto suitable support may be employed for large-scale use in the near future to solve the looming environmental crisis.The pollutant named ampicillin was almost degarded about 70 percent.

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