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Photocatalytic Degradation of Dinotefuran with Cerium Doped TiO_2

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Abstract

Organic contaminants of different classes can be degraded and completely mineralized by titania based photocatalysis. In this study, cerium-doped microporous TiO_2 crystals containing different Ce concentrations (0.3, 0.5, 0.7, and 1.0%) were synthesized using a hydrothermal method. The resulting Ce-doped TiO_2 nanoparticles were characterized using X-ray diffraction (XRD) and field emission scanning electron microscopy (FESEM). The degradation of dinotefuran was examined under artificial light (mercury lamp) in the laboratory. The effect of some parameters like pH, catalyst amount, concentration of pesticides and intensity of light were studied to achieve optimal conditions. It was observed that Ce doped TiO_2 particles exhibited higher photocatalytic activity as compared to undoped TiO_2, may be due to generation of more reactive oxidative species. The optimum conditions achieved were pH= 5.0; [Dinotefuran]= 12ppm; Ce-TiO_2=0.5% (100 mg); and Light intensity= 19.5 mwcm^{-2}. It was found that at these conditions more than 85% dinotefuran could be degraded.

***Key words*:** Cerium dopped TiO_2, Photocatalytic, Dinotefuran, Visible irradiation

The global population is rapidly expanding, and with this rise, the need for food is also escalating. To meet this growing demand, improving crop productivity is essential. Most farmers rely on pesticides to minimize the harm caused by pests and insects, aiming to protect their crops. However, the overuse of these chemicals in farming has begun to negatively impact human health and aquatic ecosystems. The unused portions of pesticides often make their way into water bodies through runoff, spray drift, leaching, and soil erosion. Consequently, both surface water and groundwater become polluted. Therefore, there is crucial need to reduced pesticides contamination by photocatalyst. Photocatalysis can play major role in pesticide degradation in presence of UV-visible light. Heterogenous photocatalytic oxidation with different semiconductor (undoped and doped), composite etc. solution of this problem.

Although titanium dioxide (TiO_2) is widely used as a photocatalyst, its large band gap restricts its activity to the ultraviolet (UV) region (wavelengths ≤ 387 nm). This limitation reduces its efficiency in practical applications. To overcome this issue, doping TiO_2 with certain metals has been explored as a strategy to reduce its band gap and extend its photocatalytic activity into the visible light range. Dinotefuran is used as neonicotinoid insecticide which is mostly used in farming to control a wide range of insects like whiteflies, aphids, thrips flea beetles bedbugs mosquitoes and leafhoppers. It works on the central nervous system of insects and disrupted to the regulatory function of nerve cells which cause paralysis and death.

The 1,1,1 trichloro-2-2-bis(p-chlorophenyl) ethane (p, p0 -DDT) is one of the most persistent pesticides. The N-doped TiO_2 was used for degradation and mineralization of the p, p0 -DDT in the presence of UV and visible light in aqueous solution. They synthesized N-doped TiO_2 nano-powder by using a modified sol-gel procedure using diethanolamine (DEA) as a source of nitrogen. It was reported that N-doped TiO_2 can effectively degrade p, p0 -DDT leading to its almost complete degradation using N-doped TiO_2 catalyst under visible light. It was also found that rate of its degradation was six times faster under visible light as compared to UV light [1].

Highly water-dispersible cerium doped titania nanoparticle deposited on reduced graphene oxide (Ce-TiO_2 / RGO) by hydrothermal method was prepared. The 0.2% cerium doped titania with 10 wt% RGO exhibited higher activity in the degradation of imidacloprid and quinalphos. It was observed that hole (h^+) and $O_2^{\cdot -}$ were responsible for photocatalytic activity of composite [2]. It was observed that Ag dopant enhanced the transformation of TiO_2 from anatase to rutile form. It was reported that anatase grain size decreased a lower concentration of Ag (2-6%), while specific surface area of titania powder was increased. It was also revealed that presence

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of Ag in crystalline TiO_2 increases its photocatalytic activity in degradation of acetamiprid. It was reported that photolytic degradation was found to increase rapidly with Ag content (n = 0.02 to 0.06), while it decreases slowly by Ag content (n = 0.08 to 0.12). It was also revealed optimal % of Ag in TiO_2 should be between 4 to 8% [3].

The phosphorus-doped carbon nitride (HPCN0.5) was synthesized via hydrothermal method. While phosphorous acid added a tube-like structure observed with stacked planar sheets. HPCN0.5 showed sixfold higher degradation efficiency than bulk g-C_3N_4. The acute toxicity tests showed that toxicity of dinotefuran reduced to earthworms after treatment by HPCN0.5. HPCN0.5 also degraded three other neonicotinoids, which proved its (HPCN0.5) great potential in alleviating negative effects of neonicotinoids [4].

The co-doped (nitrogen and cobalt) TiO_2 nanoparticles were synthesized via wet impregnation methods. They used this as-prepared (N, Co)-codoped TiO_2 for the mineralization of bisphenol-A in the presence of visible light irradiation. It was observed that 1.5% Co and 0.5% N-co-doped TiO_2 exhibited higher activity than commercial TiO_2 (Degussa 25). It was reported that there was 97% removal of total organic carbon [5]. The photocatalytic degradation of acetamiprid was studied in aqueous suspensions of TiO_2 under UV irradiation in the presence of oxygen. The experiments were conducted with acetamiprid concentrations ranging from 0.5-2.0 mg L^{-1}. It was observed that the pH of the solution changed from 5.0 to 2.0 during photocatalytic process. Some intermediates detected during the degradation such as formic acid and acetic acid, acetaldehyde and 6-chloronicotinic acid which were, completely mineralized within 6 h [6].

The La-doped TiO_2 catalyst was synthesized using the sol-gel method and evaluated for its effectiveness in degrading the neonicotinoid insecticide dinotefuran (DIN). The effects of different factors (concentration of DIN, pH, input power and addition of metal ions) on the removal rate of DIN were observed and optimum conditions were achieved as: [DIN] = 100 mg L^{-1}, power = 150 W, pH = 10.5, and [Fe^{2+}] catalyst ions = 50 mg L^{-1}. It was revealed that the degradation efficiency of DIN was increased to 99.0% with 10 wt% La-TiO_2 in 3 3 h. It was also confirmed that OH radical played an important role in process of this degradation [7].

The C^-, N^-, B^- and F^- doped nano-TiO_2(CNBF- TiO_2) catalyst were prepared to control the problem of recombination of e^-/ h^+ pair. It is known that TiO_2 is inactive, but it can be made active by doping TiO_2 with some non-metals (C, N, B and F). It was used for degradation of imidacloprid (IMI) and thiamethoxam (TMX) in presence of visible light. It was observed that CNBF/TiO_2 could degrade IMI completely in 150, 240, and 330 min, while TMX was degraded completely in 210, 270 and 420 min under UV, solar and visible irradiations, respectively. The recyclability test of as-prepared composite was also carried out, which indicated its stability in photocatalytic reaction [8].

Cerium and nitrogen co-doped TiO_2 nanocrystals were synthesized using the sol-gel method, with tetrabutyltitanate serving as the titanium precursor. The photocatalytic degradation of glyphosate was examined in an aqueous suspension of the co-doped TiO_2 under visible light irradiation. The results showed that pure TiO_2 exhibited greater thermal stability in the anatase phase. However, the co-doped TiO_2 demonstrated a significantly enhanced degradation rate—approximately five times higher than that of commercial Degussa P25 [9].

The degradation of dimethoate in Bok choy under the field conditions was investigated. The cerium doped nano-titanium dioxide (TiO_2/Ce) hydrosol was used as a photocatalyst. It was reported that TiO_2 /Ce hydrosol can degrade dimethoate in Bok choy. It was revealed that application of TiO_2 /Ce hydrosol increased the reactive oxygen species (ROS) contents of the treated Bok choy, which enhanced the degradation of dimethoate [10].

Five tripoded TiO_2 photocatalysts (P-N-S/TiO_2, Fe-N-P/TiO_2, Fe-N-S/TiO_2, Fe-Pr-N/TiO_2 and Pr-N-S/TiO_2) were prepared for the removal of thiacloprid (THI) under visible, UV-A and solar light irradiation. It was observed that doped photocatalysts not only promoted photodegradation of thiacloprid under UV-A light irradiation, but it also extended the optical response of TiO_2 to visible range. As a result, degradation of THI was improved. It was revealed that Fe-N-P tridoped TiO_2 sample exhibited the highest photodegradation of THI degree as 73, 64 and 29% under solar light, visible light and UV-A light, respectively [11].

The titanium dioxide was prepared by anodizing method and then doped it by N and F to get Fe-N-TiO_2 /Ti electrode via sol-gel method. It was revealed that Fe-N-TiO_2 /Ti was formed from anatase crystal, and it has nanotube-like structure. It was observed that thiamethoxam could be degraded using TiO_2 /Ti electrode under UV light irradiation, while Fe-N-TiO_2 /Ti was active in presence of visible light irradiation [12].

The Zn^{2+} doped TiO_2 was synthesized by photodeposition method. The photocatalyst was used in the photocatalytic degradation of malathion. As-prepared nanoparticles were having anatase crystalline (12.9 nm). It was reported that Zn^{2+} doped TiO_2 synthesized using 43.0 wt% Zn exhibited the best photocatalytic activity. The optimum value of variables was found as [Malathion] 177.59 mg L^{-1}, Catalyst dose= 0.99 g L^{-1}, pH= 10.99 and reaction time =81.04 min under UV irradiation. It was found that degradation and mineralization were found to be 98 and 74%, respectively under optimized conditions [13].

The photocatalytic degradation of thiamethoxam, imidacloprid and clothianidin was observed with light in presence of titanium oxide on glass slides at 355 nm [14]. It was reported that degradation followed the order:

Imidacloprid > Clothianidin > Thiamethoxam

Photocatalysts utilize light energy to generate electron-hole pairs, which can then participate in redox reactions to degrade various organic compounds, including pesticides. To improve the photocatalytic efficiency of TiO_2, various modification strategies are being explored. Among these, doping with cerium has shown potential; however, limited research has been conducted globally on the use of cerium-doped TiO_2 for the degradation of pesticides, particularly in the case of dinotefuran.

EXPERIMENTAL SECTION

Synthesis of Ce-doped TiO_2 photocatalyst

Hydrothermal method was used for synthesis of Ce doped titania dioxide. A solution of conc. H_2SO_4 : $C_{16}H_{40}O_4$ Ti : C_2H_5OH was prepared in volume ratio (1: 10: 30) with continuously stirred for 15 min and then a clear yellow colour solution was obtained. Then glacial acetic acid was added dropwise into this solution with continuous stirring. A white precipitate was observed in the form of slurry which was sonicated for 3 h at 50-60 °C and transferred into a Teflon coated autoclave for hydrothermal treatment at 120 °C for 14 h. After cooling the autoclave to room temperature, the solid product settled at the bottom, and the mother liquor was carefully decanted. The resulting solid was washed 3-4 times with ethanol and deionized water, followed by centrifugation to remove impurities. The purified material was dried in an oven at 60 °C for 12 hours and subsequently calcined at 500 °C for 3 hours. The final product obtained was a white solid, identified as the photocatalytic nanomaterial.

A certain amount of $(NH_4)_2[Ce(NO_3)_6]$ was used to prepare 0.3, 0.5, 0.7 and 1.0% Ce doped TiO_2. The other process was similar to preparation of TiO_2.

Characterization of Cerium doped TiO_2

X-Ray diffraction (XRD)

X-Ray diffraction (Bruker d8 ADVANCE X-ray diffractometer) was used for recording XRD of the sample of Ce-doped TiO_2. The peaks were observed at 2θ value between 20° to 80°. The sharp peaks are showing that particles of cerium doped TiO_2 are highly crystalline in nature. The size of particles was determined by using Debye–Scherrer equation:

$$D = (k\lambda/\beta \cos \theta) \ldots\ldots\ldots\ldots\ldots (1)$$

Where;

D = Crystalline size, K is the Scherer's constant (K = 0.94), λ is the X-ray wavelength (1.54178Å) and β is full width at half maximum (FWHM). The average particle size of the sample was found to be is 7.51 nm. X-Ray diffraction pattern is given in (Fig 1).

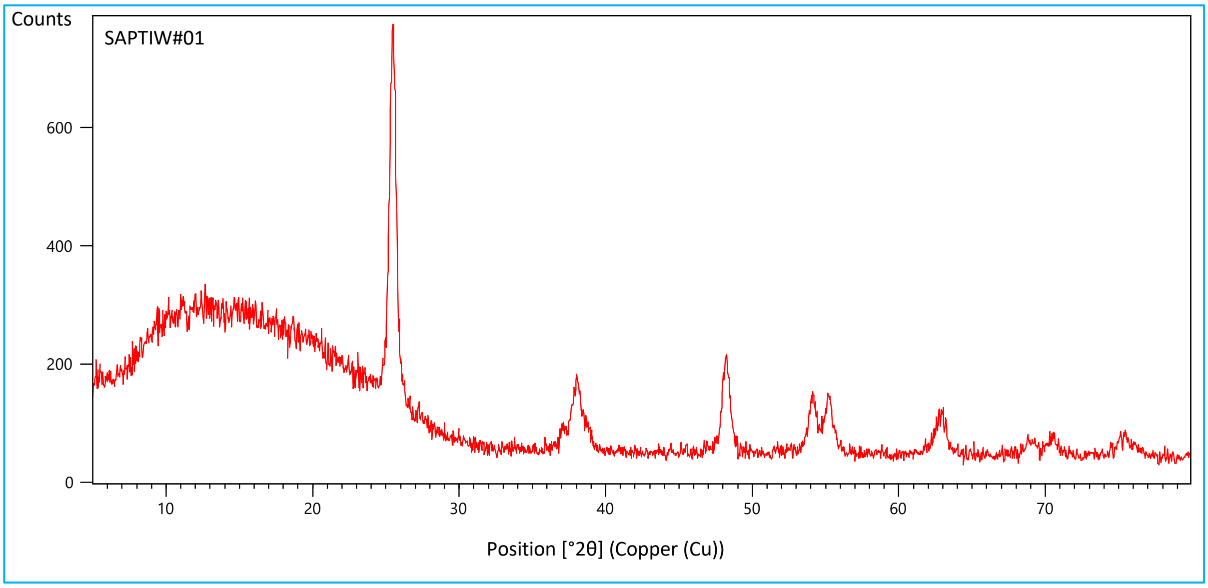

Fig 1 XRD Pattern of cerium doped TiO_2

Field emission scanning electron microscopy (FESEM)

Field emission scanning electron microscopy (FESEM) image of Cerium doped TiO_2 was recorded on Quanta200-3D FEI instrument. The Field emission scanning electron microscopy (FESEM) image of Ce-doped TiO_2 is presented in (Fig 2). The images indicated that morphology of cerium doped TiO_2 is broccoli shape or small pumice stone like. FESEM images are given in (Fig 2).

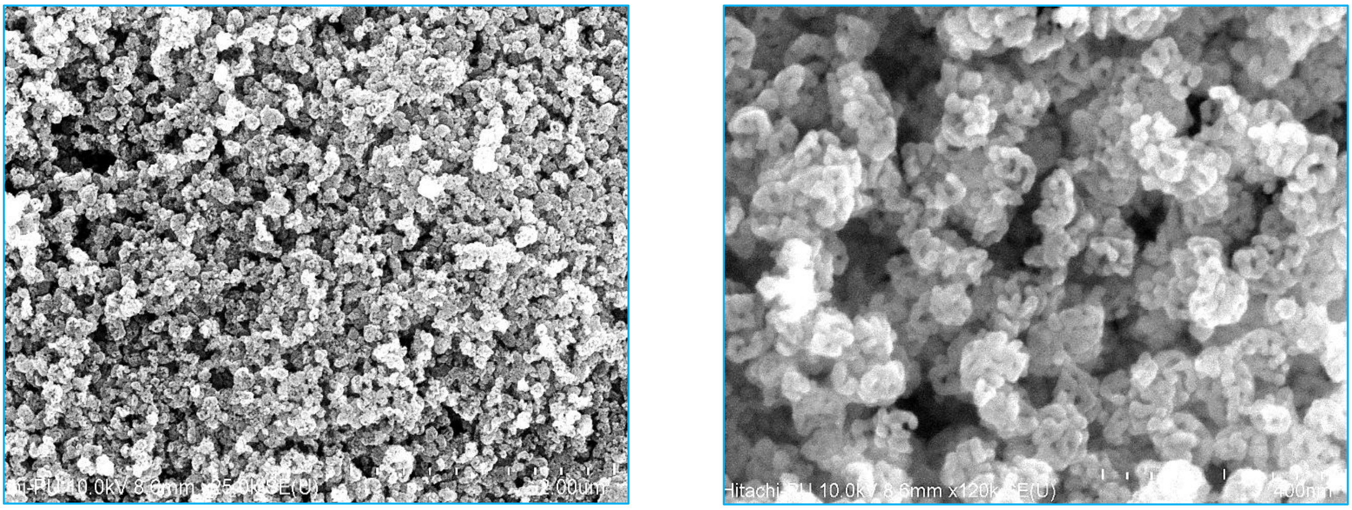

Fig 2 FESEM images of cerium doped TiO_2

Energy dispersive spectroscopy (EDS)

The cerium doped TiO_2 (Titanium dioxide) was also analyzed by Energy Dispersive Spectroscopy (EDS) to check its elemental composition. The images confirmed that cerium was present as dopant on TiO_2 (Titanium dioxide) (Fig 3) and it contains only cerium, titanium, and oxygen. It was therefore concluded that the prepared sample has no any other impurities. EDC Tecnai T-20 instrument was used for this analysis. Energy Dispersive Spectroscopy (EDS) Image is given in (Fig 3).

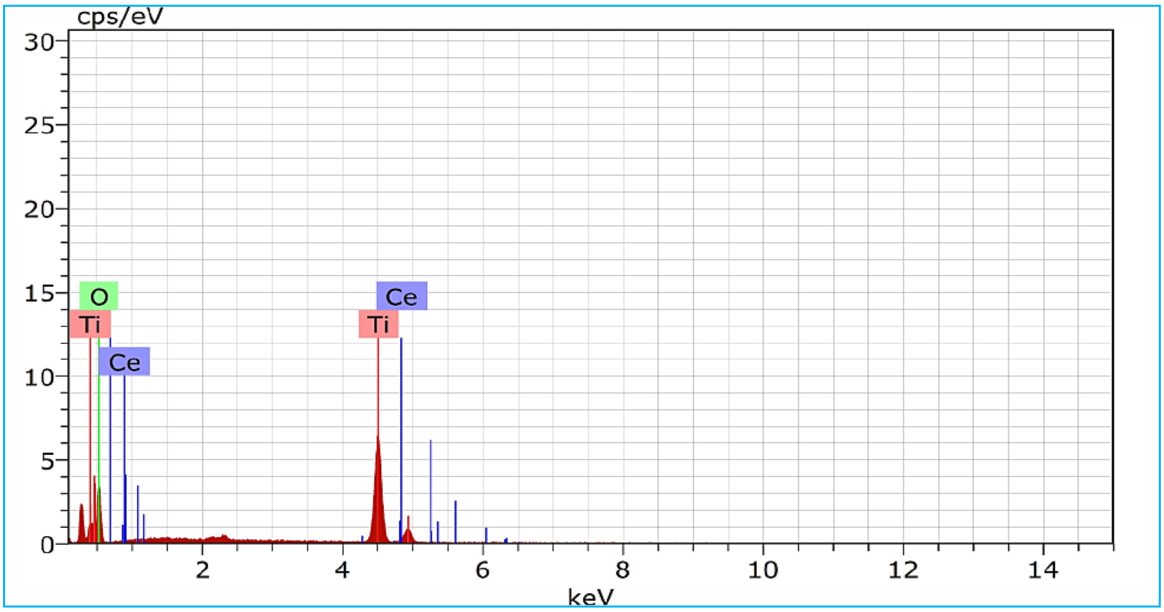

Fig 3 EDS image of cerium doped TiO_2

The following chemicals were used in present investigation: Titanium tetrabutoxide (99% SRL), ammonium ceric nitrate (99.0% Quligance), ethanol (Rankem), glacial acetic acid (Rankem) and distilled water. A 250 W medium pressure Hg vapor lamp was used for irradiation purpose A continuous water supply-maintained for restricting rise in temperature of solution. The structure of dinotefuran in (Fig 4).

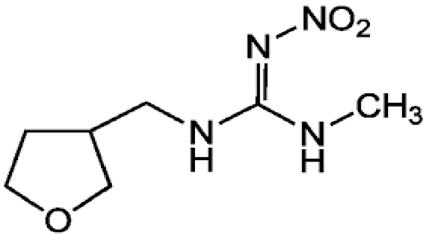

Fig 4 Chemical structure of dinotefuran

Photocatalytic experiment

The photocatalytic activity of the synthesized Ce-doped TiO_2 nanoparticles was evaluated for the degradation of dinotefuran in aqueous solution under UV–visible irradiation. A 50 mL solution of dinotefuran (12 ppm) was prepared in a 100 mL beaker. The pH of solution was maintained 5.0 by previously standardized sulfuric acid solution.

The photocatalyst (100 mg) was added into 50mL of dinotefuran solution and stirred vigorously with magnetic stirrer. The reaction mixture was then exposed to a 250 W mercury arc lamp. The suspension (solution) was then kept in the dark for 30 min to establish absorption-desorption equilibrium. After this 5 mL of the solution withdrawn, centrifuged, and filtered through a 0.22 μm membrane to remove suspended particles. The resulting filtrate was analyzed using a UV–visible spectrophotometer (Systronics Model 106) at a wavelength of 270 nm. The degradation of dinotefuran was monitored at 60-minute intervals, and after 360 minutes of irradiation, more than 85% degradation was observed. The degradation of dinotefuran % was calculated with the formula:

$$\text{Photodegradation (\%)} = \frac{A_0 - A_t}{A_0} \times 100 \quad \text{……… (2)}$$

Where A_0 = Initial Absorbance and
A_t = Absorbance at different time intervals

A progressive decrease in absorbance was observed with increasing exposure time, which indicated that dinotefuran is degraded photocatalytically in the presence of Ce-TiO_2 (Titanium dioxide).

A plot between log absorbance versus time was found to be linear which indicated that the reaction followed pseudo first order kinetics (Table 1). The rate of constant for this degradation was calculated by the relation.

$$k = 2.303 \times \text{slope} \quad \text{…………..… (3)}$$

It was also confirmed that this degradation does not possible in absence of either light or Ce-TiO_2, which confirms that this reaction is photocatalytic in nature and not photochemical or thermal reaction. The highest rate of dinotefuran contaminated aqueous solution was observed with 0.5% Ce doped TiO_2 (Titanium dioxide).

Table 1 typical run

Time (min)	Absorbance (A)	2+ log A
0	0.959	1.98
60	0.685	1.84
120	0.437	1.64
180	0.329	1.52
240	0.182	1.26
300	0.112	1.05
360	0.063	0.80

The optimum rate constant (k) was $1.26 \times 10^{-4} s^{-1}$

Effect of cerium dopant

In this experiment, photodegradation of aqueous solution of dinotefuran was performed by using undoped TiO_2 (Titanium dioxide) and ratio of cerium doped TiO_2 (Titanium dioxide) (0.3%, 0.5%, 0.7% and 1.0%). Results are reported below in (Table 2).

Table 2 Effect of cerium dopant

Photocatalyst	Rate Constant (k) × $(10)^4$
Undoped TiO_2	0.33
0.3 % Ce doped TiO_2	0.85
0.5 % Ce doped TiO_2	0.99
0.7 % Ce doped TiO_2	0.90
1.0 % Ce doped TiO_2	0.68

It was observed that 0.5 % cerium dopant was shown highest rate of degradation.

The effect of pH

The pH of the solution is an important factor that can significantly influence the rate of degradation. Experimental results indicated that the highest degradation rate occurred at a pH of 5.0 (Table 3). The surface charge of the photocatalyst can be either positive or negative, depending on the pH of the system. Under acidic conditions, the catalyst surface becomes positively charged, while in alkaline conditions, it acquires a negative charge. In this study, the pH was adjusted within the range of 4.0 to 9.0 using dilute sulfuric acid and sodium hydroxide (NaOH) solutions (Table 3).

Table 3 Effect of pH

pH	Rate constant (k) × $(10)^4$
4.0	0.94
5.0	1.26
6.0	1.06
7.5	0.99
8.0	0.89
9.0	0.75

The effect of dinotefuran concentration

The concentration of dinotefuran may also affect the rate of photodegradation. The results are represented in (Table 4). As substrate concentration was increased from 2 ppm to 15 ppm the rate of degradation increases up to 12ppm, but there was a decrease on increasing the concentration after further. The maximum degradation occurred at a concentration of 12 ppm (Table 4).

Table 4 Effect of dinotefuran concentration

Dinotefuran concentration	Rate constant (k) × $(10)^4$
2	0.72
4	0.74
6	0.80
8	0.86
10	1.02
12	1.26
15	1.04

Effect of amount of photocatalyst

The effect of amount of photocatalyst (Ce-TiO_2) was observed on rate of degradation of dinotefuran using 0.02 - 0.12 g. The results are given in (Table 5). The photocatalytic degradation rate was observed to increase initially up to 0.1g and then it began to decrease. This increase may be due to increasing in number of active sites (depending upon exposed surface area). Then degradation rate decreases because of agglomeration of semiconductor nanoparticles, which reduces the effective surface area available for reaction (Table 5).

Table 5 Effect of amount of photocatalyst

Concentration in ppm	Rate constant (k) × $(10)^4$
20	0.15
40	0.56
60	0.97
80	1.09
100	1.26
120	1.06

Effect of light intensity

The distance of source was varied to change the light intensity in the range of 13.36 mW cm^{-2} to 23.7 mW cm^{-2}. The results are reported in (Table 6) and the highest degradation rate was observed at light intensity, 19.2 $mWcm^{-2}$.

The results in (Table 6) clearly demonstrate that the rate constant (*k*) is strongly influenced by light intensity, showing a nonlinear relationship rather than a simple proportional increase. At the highest tested intensity (23.7 mW cm^{-2}), the rate constant is moderate (1.04×10^{-4}), but interestingly, at a slightly lower intensity of 19.2 mW cm^{-2}, the rate constant reaches its maximum value (1.26×10^{-4}), suggesting the presence of an optimal light intensity for the process. Beyond this optimum, the rate constant declines, as observed at 15.9 and

13.4 mW cm^{-2} (0.93 and 0.89 × 10^{-4}, respectively), indicating that reduced photon availability limits the excitation of electrons and the generation of reactive species necessary for sustaining the reaction. Conversely, at very high intensity, excess photons may lead to recombination losses of photogenerated charge carriers or inefficient utilization of energy, preventing further enhancement of reaction kinetics. This pattern implies that the system achieves maximum efficiency at an intermediate light intensity, where the balance between photon absorption and charge carrier utilization is optimal. Such findings are particularly important in photocatalytic or photochemical processes, emphasizing that simply increasing light intensity does not guarantee higher reaction rates; instead, careful optimization is essential for achieving both efficiency and energy economy in practical applications.

Table 6 Effect of light intensity

Light intensity (mW cm^{-2})	Rate constant (k) × $(10)^4$
23.7	1.04
19.2	1.26
15.9	0.93
13.4	0.89

Scavenger test

A significant reduction in the degradation rate was observed in the presence of isopropyl alcohol, indicating that hydroxyl radicals played a key role as the main oxidizing agents in the degradation mechanism.

Mechanism

$$ {}^{1}DNT_{0} \xrightarrow{h\upsilon} {}^{1}DNT_{1} \quad \ldots\ldots\ldots\ldots\ldots\ldots\ldots (4)$$

$$ {}^{1}DNT_{1} \xrightarrow{h\upsilon} {}^{3}DNT_{1} \quad \ldots\ldots\ldots\ldots\ldots\ldots\ldots (5)$$

$$ SC \xrightarrow{h\upsilon} e^{-}(CB) + h^{+}(VB) \quad \ldots\ldots\ldots\ldots\ldots\ldots\ldots (6)$$

$$ h^{+} + {}^{-}OH \xrightarrow{} {}^{\bullet}OH \quad \ldots\ldots\ldots\ldots\ldots\ldots\ldots (7)$$

$$ {}_{3}DNT^{1} + {}^{\bullet}OH \xrightarrow{} Products \quad \ldots\ldots\ldots\ldots\ldots\ldots\ldots (8)$$

Where;
DNT = Dinotefuran
SC = Semiconductor (Ce-TiO_2)

CONCLUSION

In the absence of cerium doping, the photocatalytic degradation efficiency of dinotefuran was limited to 50%, which is significantly lower than that achieved with cerium-doped TiO_2. The optimum values of operating parameters were determined as pH = 5.0, concentration of dinotefuran = 12 ppm, amount of Ce-TiO_2 = 100 mg, and light intensity = 19.2 m Wcm^{-2}. Under these optimized parameters, more than 85% of dinotefuran was successfully degraded.

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