

Enhanced photocatalytic degradation of sulfadiazine by Fe^{3+} in aqueous TiO_2 suspension

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Abstract: In order to explore the reaction mechanism of Fe^{3+} and the mineralization effect of the micropollutant, Fe^{3+} -assisted photocatalytic oxidation of sulfadiazine (SD) in the TiO_2 suspended solution is investigated. The effect of Fe^{3+} participation, the degradation kinetics of SD, the effect of SD mineralization and the possible mechanism of Fe^{3+} participation in TiO_2 suspension are analyzed by adding FeCl_3 , taking samples at a given time and determining the SD concentration. Results indicate that the degradation of SD catalyzed by $\text{TiO}_2/\text{Fe}^{3+}$ is faster than that catalyzed by TiO_2 or Fe^{3+} separately. The photocatalytic degradation of SD follows the pseudo-first-order kinetics model in a range of 20 to 80 mg/L of initial concentration. The mineralization rate of SD can be enhanced by the addition of Fe^{3+} in the TiO_2 suspended solution. The mechanism of the rapid degradation of SD is proposed, which indicates that Fe^{3+} adsorbed on the surface of TiO_2 particles acts as an electron acceptor. The amount of recombining electron-hole pairs decreases, and the amount of hydroxyl radicals increases. The increased hydroxyl radical strengthens the degradation of SD in the $\text{TiO}_2/\text{Fe}^{3+}$ suspended solution.

Key words: TiO_2 ; photocatalytic degradation; sulfadiazine; Fe^{3+} ; enhancement

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As a subclass of pharmaceuticals, sulfonamides (SAs) are commonly used in both human therapy and veterinary husbandry. SAs have received increasing attention due to the possibility of bacterial resistance in recent years^[1-2], and SAs have been detected in groundwater, treated water effluents and reclaimed water due to discarded drugs, animal manure and the manure waste lagoons^[3-4]. Therefore, there is a growing interest in removing SAs from water. Although past studies demonstrate that SAs are biodegraded in sewage, this process is typically too slow for the wastewater effluent^[5]. Ternes et al.^[6] reported that due to the low tendency of sulfonamide to accumulate in sludge, the removal rate of sulfonamide is 24% after 16 d retention time in the effluent of the Braunschweig wastewater treatment plant.

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Sulfadiazine (SD) as one of the SAs is widely used in aquaculture and animal husbandry, and it has been detected at high frequencies^[7-9]. Therefore, it is necessary to develop an effective technology system to remove the sulfonamides in aqueous environments.

Semiconductor photocatalysis is a new promising method for the removal of environmental pollutants in water. The organic pollutants can be decomposed by the participation of hydroxyl radicals which are generated on the surface of semiconductor. This process is initiated when the photos with energy are equal to or greater than the band gap energy of photocatalyst. Unfortunately, there is an electron-hole recombination step which results in a low yield of hydroxyl radicals^[10]. Thus, it is very important to prevent the recombination of the electron-hole. TiO_2 is considered to be a suitable semiconductor for the environmental photocatalytic application, and also there is an electron-hole recombination in TiO_2 particle^[11]. Therefore, the most important task for TiO_2 photocatalysis is to reduce the recombination of the electron-hole. Some excellent methods, such as noble metal deposition, ion-doping, dye sensitization and surface reduction treatment, are adopted to improve the performance of TiO_2 ^[12]. However, these methods require a complicated process or a series of chemical events. The Fe^{3+} self-loading technique has attracted interest in recent years^[13], and the degradation of organic pollutant can be improved by the presence of Fe^{3+} in the TiO_2 suspended solution. However, the published papers focus on the simple description of rapid degradation^[13-14], and it falls short of systematic research on the role of Fe^{3+} and the mechanism of rapid degradation of organic pollutants in the TiO_2 suspended solution. Furthermore, to the best of our knowledge, there are few studies to analyze the degradation kinetics, to explore the participation mechanism of Fe^{3+} , and to examine the mineralization effects of organic pollutants.

In this paper, a systematic research is performed in order to study the interaction mechanism between TiO_2 and Fe^{3+} and to analyze the degradation kinetics of the micropollutant with sulfadiazine (SD) as a model compound. SD is selected as the model pollutant in this paper since it is a typical antibiotic widely used in aquaculture and animal husbandry. More importantly, in this paper, we investigate the rapid degradation of antibiotics and improve the performance of the catalyst (TiO_2) by excluding further treatment.

1 Experimental

1.1 Materials

Sulfadiazine (99.9% purity, Fig. 1) is purchased from

Yuancheng Group (China). TiO_2 P-25 (anatase: rutile is 3.6:1; the particle size is 21 nm, and the surface area is $(50 \pm 15) \text{ m}^2/\text{g}$) is purchased from Degussa Inc. (Germany). $\text{FeCl}_3 \cdot 6\text{H}_2\text{O}$ used as the catalyst is purchased from Country Medicine Reagent Co. (China). HPLC grade Acetonitrile is from Shanghai Lingfeng Chemical Reagent Co. (China). Other chemicals are analytic reagent grade (Country Medicine Reagent Co., China). N_2 gas (99% purity) is from Wenda Co. (China).

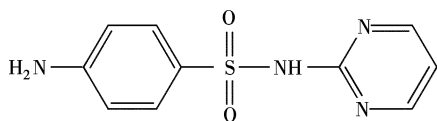
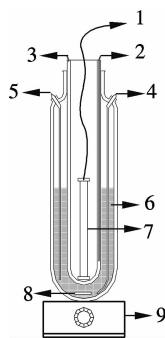


Fig. 1 Structure of sulfadiazine ($\text{C}_{10}\text{H}_{10}\text{N}_4\text{O}_2\text{S}$, CAS 68-35-9)

1.2 Procedure

The experiments are carried out in a cylindrical reactor (1 000 mL capacity, Nanjing Xujiang Co., China, see Fig. 2) with a 15 W medium pressure mercury light at a wavelength (λ_{max}) of 365 nm. The outside part of the reactor device is a thimble which contains the reaction solution, and the inside part of the reactor device is a thimble which maintains the reaction temperature (25°C) by running water. The lamp is placed in a cooling trap, and its incident photo flux is $1.32 \times 10^{-4} \text{ Einstein}/(\text{L} \cdot \text{min})^{[15]}$. The SD solutions containing an appropriate dosage of TiO_2 powder and/or Fe^{3+} are prepared in the dark. The pH value of the solution is controlled at 3.5 by HCl and NaOH^[13-14]. In order to reach the adsorption equilibration, the solution is stirred for 30 min in the dark. At different intervals, the samples are withdrawn and centrifuged at 4 000 r/min for 30 min, and the supernatant of the samples are determined by HPLC. All the solutions are prepared in reagent-grade deionized water.



1—Electrical source; 2—Cooling water in; 3—Cooling water out; 4—Gas in; 5—Sample; 6—Solution; 7—Lamp; 8—Rotor; 9—Magnetic stirrer

Fig. 2 Schematic diagram of photochemical reaction device

1.3 Analysis

The concentration of SD is determined by HPLC (L6, Pgrandsil STC C18 column [$4.6 \text{ mm} \times 150 \text{ mm}$, $5 \mu\text{m}$], Pgenenal Co., China) with a flow rate of $1.0 \text{ mL}/\text{min}$ and a UV detector (UV6) at 285 nm. The mobile phase is a mixture of acetonitrile and $0.02 \text{ mol}/\text{L}$ phosphoric acid (The volume ratio is 25/75). The ions in the solution are detected by a DIONEX-DX 100 ANIONS-CATIONS ana-

lyzer (Dionex, USA). The total organic carbon (TOC) is determined by the total carbon analyzer (Shimadzu 5 000 A, Japan), and the mineralization rates of SD for samples are calculated by

$$\eta = \frac{\text{TOC}_0 - \text{TOC}_t}{\text{TOC}_0} \times 100\% \quad (1)$$

where η is the mineralization rate of SD; TOC_0 and TOC_t represent the amount of total organic carbon after 0 min and t min irradiation, respectively.

2 Results and Discussion

2.1 Degradation experiments of SD

There is almost no change in the concentration of SD in absence of catalysts in the dark and the direct photolysis of SD by UV is very slow (see Fig. 3). In contrast, the degradation of SD is fast in the presence of TiO_2 or Fe^{3+} in the solution, and the degradation rate of SD by TiO_2 photocatalysis is higher than that by Fe^{3+} photocatalysis. The difference between TiO_2 and Fe^{3+} may arise from different ways of generation of hydroxyl radicals. Fe^{3+} aqua-complexes may absorb the UV light to generate the hydroxyl radical, and $\text{Fe}(\text{OH})^{2+}$ is the main photoreactive species^[16]. However, the formation of $\text{Fe}(\text{OH})^{2+}$ produces H^+ which can result in the pH change of the reaction solution^[17], and the yield of the hydroxyl radical decreases since the concentration of $\text{Fe}(\text{OH})^{2+}$ is influenced by the pH change of the solution. Thus, the degradation rate of SD is greatly influenced by the non-stable photocatalytic process.

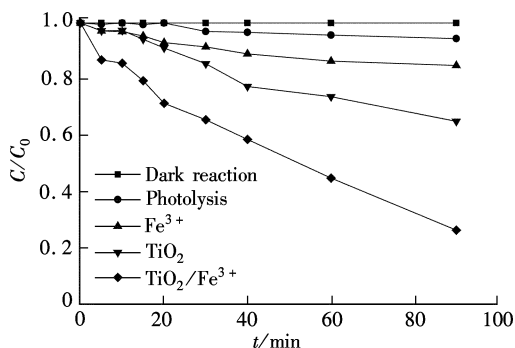


Fig. 3 Concentration change of SD vs. time in degradation experiments ($\rho(\text{SD}_0) = 50 \text{ mg}/\text{L}$, $c(\text{Fe}_0^{3+}) = 0.1 \text{ mmol}/\text{L}$, $\rho(\text{TiO}_2) = 0.5 \text{ g}/\text{L}$, pH = 3.5, the gas flow (air) is $0.5 \text{ L}/\text{min}$)

As for TiO_2 , the hole h_{VB}^+ and the electron e_{CB}^- are generated in the valence band and the conduction band of TiO_2 , respectively, under UV irradiation^[11]. A part of electrons are directly transferred to the molecule adsorbed on the surface of TiO_2 particles, and the positive hole can oxidize the hydroxide ion adsorbed on the surface of TiO_2 particles to produce the hydroxyl radical. TiO_2 has a better stability than Fe^{3+} in solution^[18], thus the degradation rate of SD by TiO_2 photocatalysis is higher than that by Fe^{3+} photocatalysis.

The results also show that the degradation of SD catalyzed by $\text{TiO}_2/\text{Fe}^{3+}$ is faster than that by TiO_2 photocatalysis or Fe^{3+} photocatalysis. About 74% of SD is degraded by $\text{TiO}_2/\text{Fe}^{3+}$ photocatalysis after 90 min irradiation, whereas 15.1% and 35.0% of SD are degraded by Fe^{3+} photocatal-

ysis and TiO_2 photocatalysis after 90 min irradiation, respectively. The degradation rate of SD catalyzed by $\text{TiO}_2/\text{Fe}^{3+}$ is higher than that of the simple sum of single photocatalysis (TiO_2 and Fe^{3+}), which indicates that the photocatalytic degradation of SD is strengthened by the addition of Fe^{3+} in the TiO_2 suspended solution. The rapid degradation of SD by $\text{TiO}_2/\text{Fe}^{3+}$ photocatalysis may arise from the combined effect of two photocatalysts in suspension^[14], and the part of Fe^{3+} may act as the electron acceptor on the surface of TiO_2 particles.

2.2 Studies of SD mineralization

In order to investigate the degradation effects of SD, the mineralization rates of SD catalyzed by three systems (Fe^{3+} , TiO_2 and $\text{TiO}_2/\text{Fe}^{3+}$) are investigated in detail, and the results are shown in Fig. 4. The mineralization rate of SD catalyzed by $\text{TiO}_2/\text{Fe}^{3+}$ is higher than that by TiO_2 or Fe^{3+} photocatalysis, indicating that $\text{TiO}_2/\text{Fe}^{3+}$ is effective in mineralizing SD (About 23% of SD is mineralized from the suspension by $\text{TiO}_2/\text{Fe}^{3+}$ photocatalysis after 90 min irradiation). The rapid mineralization of SD catalyzed by $\text{TiO}_2/\text{Fe}^{3+}$ may arise not only from the combination effects of two photocatalysts (TiO_2 and Fe^{3+}), but also Fe^{3+} as an electron acceptor on the surface of TiO_2 particles.

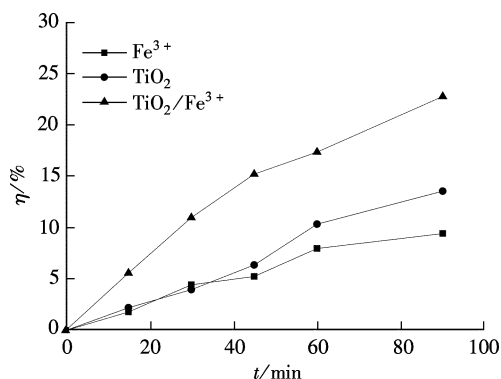


Fig. 4 Mineralization rates of SD along irradiation time ($\rho(\text{SD}_0) = 50 \text{ mg/L}$, $c(\text{Fe}_0^{3+}) = 0.1 \text{ mmol/L}$, $\rho(\text{TiO}_2) = 0.5 \text{ g/L}$, $\text{pH} = 3.5$, the gas flow (air) is 0.5 L/min)

Furthermore, the mineralization process of SD is analyzed by detecting the concentrations of two inorganic ions (SO_4^{2-} and NO_3^-). The concentrations of two inorganic ions increase with the prolongation of the reaction time, indicating that the organic sulfur and the organic nitrogen are gradually transformed into the inorganic nitrogen NO_3^- and inorganic sulfur SO_4^{2-} , respectively. After 90 min irradiation, the concentration of SO_4^{2-} and NO_3^- are 1.04 and 0.79 mg/L in the $\text{TiO}_2/\text{Fe}^{3+}$ suspension, respectively, while the concentrations of SO_4^{2-} and NO_3^- are 0.24 and 0.19 mg/L in the Fe^{3+} solution, and the concentrations of SO_4^{2-} and NO_3^- are 0.35 and 0.28 mg/L in the TiO_2 suspension, respectively (see Fig. 5). This phenomenon shows that Fe^{3+} can strengthen the mineralization of antibiotics in the TiO_2 suspension. Moreover, the difference in the concentrations of SO_4^{2-} and NO_3^- may arise from different numbers of S and N in the SD molecule.

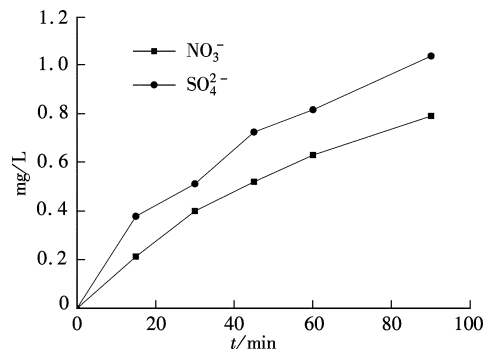


Fig. 5 Concentration changes of SO_4^{2-} and NO_3^- vs. time in degradation experiments ($\rho(\text{SD}_0) = 50 \text{ mg/L}$, $c(\text{Fe}_0^{3+}) = 0.1 \text{ mmol/L}$, $\rho(\text{TiO}_2) = 0.5 \text{ g/L}$, $\text{pH} = 3.5$, the gas flow (air) is 0.5 L/min)

2.3 Degradation kinetics of SD

The degradation kinetics is also studied by analyzing the change of concentrations. According to Refs. [11–12], the photocatalytic degradation of the organic compound in the TiO_2 suspended solution can be described by pseudo-first kinetics, which is rationalized in terms of the Langmuir-Hinshelwood model (the L-H model),

$$r_0 = -\frac{dC}{dt} = \frac{kKC_0}{1 + KC_0} \quad (2)$$

$$\frac{1}{r_0} = \frac{1}{kK} \frac{1}{C_0} + \frac{1}{k} \quad (3)$$

where r_0 is the initial degradation rate of SD; C_0 is the equilibrium bulk-solute concentration; k and K are the rate constant and the adsorption coefficient, respectively.

The concentration of SD in the range of 20 to 80 mg/L is investigated in detail. The experimental data obtained from the first 20 min of reaction time are used in calculating the initial reaction rate in order to minimize the competitive effects of intermediates^[18]. The degradation of SD catalyzed by $\text{TiO}_2/\text{Fe}^{3+}$ is found to follow the pseudo-first order kinetics well (see Fig. 6), and there is a good linear relationship between plot $1/r_0$ vs. $1/C_{\text{Eq}}$ (Inset in Fig. 6). According to the linear fit of the curve, the L-H model can be expressed as

$$\frac{1}{r_0} = 10.17 \times \frac{1}{C_0} + 1.19, \quad R^2 = 0.997 \quad (4)$$

The rate constant k and the adsorption coefficient K are $0.84 \text{ mg/(L} \cdot \text{min)}$ and 0.12 L/mg , respectively.

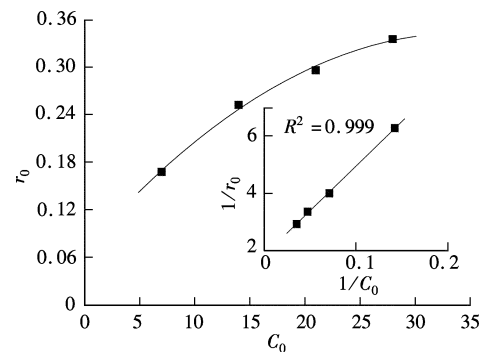


Fig. 6 Plot of r_0 vs. C_0 at different initial concentrations of SD (Inset shows transform of $1/r_0$ vs. $1/C_0$, $\rho(\text{TiO}_2) = 0.5 \text{ g/L}$, $c(\text{Fe}_0^{3+}) = 0.1 \text{ mmol/L}$, $\text{pH} = 3.5$, the gas flow (air) is 0.5 L/min)

Furthermore, the degradation kinetics of SD is well described by the L-H model, which indicates that the hydroxyl radical generated by TiO_2 (both $^1\text{O}_2$ and Fe^{3+} as the electron acceptors) may play the main role in the reaction, and the hydroxyl radical generated by Fe^{3+} itself may be lower.

2.4 Mechanism of enhanced photocatalysis

In order to prove that Fe^{3+} is the electron acceptor on the surface of TiO_2 particles, the degradation experiments of SD are carried out with N_2 as a gas medium. As shown in Fig. 7, the degradation rate of SD greatly decreases with N_2 as a gas medium in the TiO_2 suspended solution or in the $\text{TiO}_2/\text{Fe}^{3+}$ suspended solution. However, the degradation rate of SD is not influenced by N_2 as a gas medium in Fe^{3+} solution. This phenomenon is due to lack of the electron acceptor on the surface of TiO_2 particles^[11], the electron-hole can recombine very easily in the absence of oxygen, and, thus, the yield of hydroxyl radicals is greatly influenced. On the contrary, Fe^{3+} can act as the electron acceptor on the surface of TiO_2 particles with N_2 as a gas medium in suspension; thus, the hydroxyl radical is continuously generated.

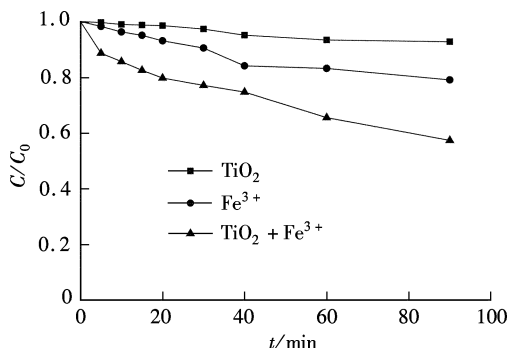
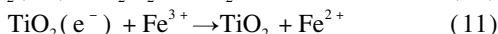
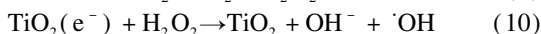
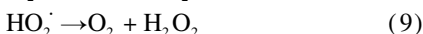
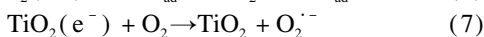
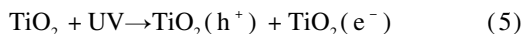
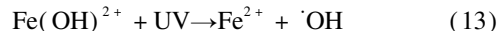
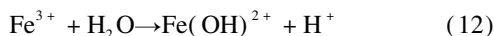


Fig. 7 Effect of N_2 on the degradation of SD ($\rho(\text{SD}_0) = 50 \text{ mg/L}$, $c(\text{Fe}_0^{3+}) = 0.1 \text{ mmol/L}$, $\rho(\text{TiO}_2) = 0.5 \text{ g/L}$, $\text{pH} = 3.5$, the gas flow (N_2) is 0.5 L/min)

The results indicate that Fe^{3+} can enhance the degradation rate of SD, that the degradation kinetics of SD follows the L-H model, and that Fe^{3+} acts the electron acceptor on the surface of TiO_2 particles. Thus, the mechanism of Fe^{3+} -assisted photocatalytic degradation of SD in the TiO_2 suspension is proposed here, which indicates that both oxygen and Fe^{3+} act as the electron acceptors to trap the electron on the surface of TiO_2 particles (see Fig. 8), and two electron trappers can improve the separation of the electron-hole, thus the yield of hydroxyl radicals is enhanced,



Meanwhile, the SD molecule is oxidized by the hydroxyl radical generated by $\text{Fe}(\text{OH})^{2+}$,



Therefore, separation of the electron-hole is a crucial factor in affecting the photocatalytic efficiency of TiO_2 ,

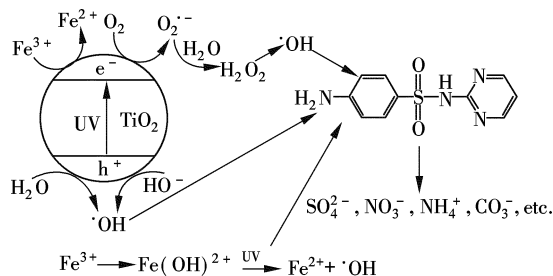
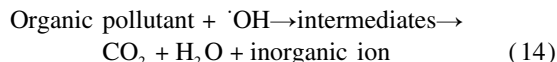


Fig. 8 Mechanism of enhanced photocatalytic degradation of SD

3 Conclusion

Under the UV lamp irradiation, Fe^{3+} -assisted photocatalytic degradation of SD in the TiO_2 suspended solution is investigated. Results indicate that the degradation kinetics of SD follows the Langmuir-Hinshelwood model, and the reaction rate constant is $0.84 \text{ mg}/(\text{L} \cdot \text{min})$. Furthermore, the mineralization of SD catalyzed by $\text{TiO}_2/\text{Fe}^{3+}$ is faster than that by TiO_2 or Fe^{3+} separately, and the concentrations of SO_4^{2-} and NO_3^- increase with the reaction time. The mechanism of the enhanced photocatalysis of TiO_2 arises from Fe^{3+} adsorbed on the surface of TiO_2 particle as the electron acceptor. The amount of recombining electron-hole pairs decreases and the amount of hydroxyl radicals increases. The increased hydroxyl radical strengthens the degradation of SD in $\text{TiO}_2/\text{Fe}^{3+}$ suspension.

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Fe^{3+} 强化 TiO_2 光催化降解磺胺嘧啶

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摘要:为了分析 Fe^{3+} 参与 TiO_2 的反应机理及微污染物的矿化效果,研究了 Fe^{3+} 辅助 TiO_2 光催化氧化降解磺胺嘧啶(SD). 通过 TiO_2 悬浮液中预添加 FeCl_3 和既定的时间取样并测定 SD 浓度的方式,分析了 Fe^{3+} 参与 TiO_2 催化降解反应后的效能、SD 的降解反应动力学和 SD 的矿化效果,探讨了溶液中 Fe^{3+} 与 TiO_2 之间可能存在的作用机理. 结果表明,光催化 $\text{TiO}_2/\text{Fe}^{3+}$ 降解 SD 的效果远好于单独使用 TiO_2 或 Fe^{3+} 催化降解时的效果. SD 的初始浓度在 20 ~ 80 mg/L 时,其降解过程遵守准一级降解动力学模型. TiO_2 悬浮液中预添加 Fe^{3+} 能够提高 SD 的矿化率. Fe^{3+} 参与 TiO_2 光催化降解反应的机理可能是部分 Fe^{3+} 承担着 TiO_2 的电子受体,降低了 TiO_2 的电子-空穴复合的程度,因而增加了溶液中羟自由基的产率,而羟自由基产率增加强化了溶液中 SD 的降解.

关键词: TiO_2 ; 光催化; 磺胺嘧啶; Fe^{3+} ; 强化

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