Review Article

Development and Application of TiO_2 Nanoparticles Coupled with Silver Halide

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Received 26 June 2014; Revised 23 September 2014; Accepted 15 October 2014; Published 6 November 2014

Academic Editor: Xuebo Cao

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Titanium dioxide (TiO_2) is proposed to be effective photocatalyst for wastewater treatment, air purification, and self-cleaning ability, because of its strong oxidation and superhydrophilicity. In order to conquer the limits of TiO_2 , a variety of methods have been used. This paper presents a critical review of novel research and achievements in the modification of TiO_2 nanoparticles with silver halide (AgX , $\text{X} = \text{Cl}, \text{Br}, \text{I}$), which aims at enhancing the visible light absorption and photosensitivity. Herein we study the synthesis, physical and chemical properties, and the mechanism of this composite photocatalyst.

1. Introduction

Water is one of the most essential substances for all life. However, an increasing number of organic pollutants are being discharged into aqueous environment along with wastewater. Various treatment techniques and processes have been used to remove the organic pollutants from wastewater. Among all the approaches proposed, the photocatalysis technology for the treatment of contaminated water has attracted significant attention in the past few decades, because of its high stability, low cost, and nontoxicity toward both humans and the environment [1]. Among a variety of photocatalysts, TiO_2 is considered as one of the most ideal semiconductors for photocatalyst. But there are some drawbacks which have greatly limited the application of TiO_2 : one is the inefficient utilization of solar energy, and the other is the rapid recombination rate of photogenerated electron-hole pairs [2].

In response to these deficiencies, several approaches for TiO_2 modification have been developed: doping by metals (noble metals, transition metals, lanthanide metals, alkaline and alkaline earth metals, cadmium sulphide, etc.) and non-metals (nitrogen, fluorine, sulfur, carbon, etc.) and modification with dye-sensitization and composite semiconductors.

After the phenomenon of surface plasmon resonance (SPR) was discovered, a novel photocatalyst named plasmonic photocatalyst was developed, which was a composite

photocatalyst composed of noble-metal nanoparticles and a polar semiconductor, such as silver halide (AgX). In the photographic process, an electron is liberated by photon adsorption and electron-hole pair creation. The liberated electron will combine with an Ag^+ cation to form an Ag^0 atom. Continuous irradiation will bring limitless photons, which will be absorbed by the AgX particles to form a cluster of Ag^0 atoms, and this phenomenon is the very basis for chemical photography. Considering its instability under sunlight, AgX is scarcely ever used as a photocatalyst. In the study of the photocatalyzed H_2 production from a CH_3OH aqueous solution in the presence of AgBr dispersed on a silica support, Kakuta et al. observed that Ag^0 atoms take place on the surface of AgBr particles and AgBr particles are not destroyed under UV illumination [3]. This research leads to the synthesis of the first visible light plasmonic photocatalyst Ag/AgCl . Similarly, the combination of AgX and TiO_2 can also have this kind of property. The Ag/AgCl ought to have both advantages, which can be excited under visible light irradiation and can be stable when illuminated.

2. TiO_2 Coupled with Silver Halide

2.1. AgCl/TiO_2 . With a direct band gap of 5.6 eV and an indirect band gap of 3.25 eV, silver chloride (AgCl) is widely

acknowledged as photosensitive material and is one of the source materials of photographic films [4]. Since 2008, Wang et al. reported the synthesis of a highly efficient and stable plasmonic photocatalyst Ag/AgCl under visible light irradiation through treating Ag_2MoO_4 with HCl to form AgCl and subsequently reducing some Ag^+ to Ag^0 nanoparticles on the AgCl surface by illumination. Taisuke et al. prepared Ag(core)-AgCl(shell) composite crystal-loaded TiO_2 by a two-step method involving electrochemical reduction/electrochemical oxidation. They showed that an energy gap of more than 0.7 eV is needed for efficient one-directional interfacial electron transfer from the CB (conducting band) edge of TiO_2 to the redox potential of Ag/AgCl [5]. It was reported that TiO_2 nanotubes were first synthesized by hydrothermal method. Since then, they had been widely used in photocatalysis [6]. Yu et al. fabricated visible-light-driven plasmonic photocatalyst Ag/AgCl/ TiO_2 nanotubes (NTs) by AgCl nanoparticles doping into the self-organized TiO_2 NTs which were grown by anodization of Ti foils in an aqueous solution. They also revealed the reason why the photoexcited electrons at Ag NPs can inject into the TiO_2 conduction band is that a Schottky barrier is formed at the Ag/ TiO_2 interface and the electric field in the space-charge layer was able to promote transport of excited electrons at the Ag surface to the walls of TiO_2 NTs [7]. Wen and Ding used AgCl nanoparticles to load onto the TiO_2 nanotubes by a precipitation reaction, and some AgCl nanoparticles were reduced to Ag particles under irradiation. The product Ag@AgCl/ TiO_2 nanotubes exhibited high photocatalytic activity due to the surface plasmon resonance (SPR) effect [8]. Two-step method consisting of electrochemical anodization process and electrodeposition process was used to prepare the Ag/AgCl/ TiO_2 nanotube arrays. It was found that the major reactive species produced in situ are h^+ , $\cdot\text{OH}$, and O^{2-} via the degradation of microcystin LR. The results showed that the degradation reaction was highly promoted in NaCl electrolyte, as well as in an acid solution [9].

The composite also can be made into thin films. Zhou et al. fabricated the Ag/AgCl/ TiO_2 nanocomposite thin films by a sol-gel and photochemical reduction method. The samples show high visible light photocatalytic activity for degradation of 4-chlorophenol aqueous solution and recycling stability [10].

Chemical doping of TiO_2 with nonmetallic elements (C, N, B, S, or F) to extend their visible absorbance is a commonly used method [11, 12]. A report from Yan et al. chose C and F as codopants, which resulted in a red-shift of the absorption edge to 510 nm from 390 nm. Then AgCl was loaded to these codoped TiO_2 to enhance its photocatalytic property. The brief mechanism is proposed to the transformation of holes from C, F-codoped TiO_2 to the AgCl phase [13].

Compared with the commercial P25 photocatalyst, Sangchaya et al. [14] and their team found that TiO_2 -AgCl powders which were synthesized by a sol-gel method were more efficient under UV and visible light irradiation. In the test of photocatalytic degradation of methyl blue (MB), they found that a photocatalytic reaction rate (R) can be written as $R = 1.00e^{-kt}$ where k is a rate constant and t is a treatment

time in hour. The result showed that k value of TiO_2 -AgCl powders was 0.47 which was superior to P25's 0.12. This phenomenon indicates the photoinduced electron trapping effect of AgCl, higher concentration of OH radicals on TiO_2 -AgCl surface, and their smaller crystallite size. And the degradation of MB with TiO_2 -AgCl is 50% after 6 h visible light irradiation, while that of P25 is 32%. In some ways, coupling with AgCl can overcome the two major defects of TiO_2 .

Updating the synthesis methods or changing conditions of the degradation progress can not only increase the photocatalytic activity, but also reveal the mechanism, although there are still some details in the reaction that remain unclear.

2.2. AgBr/ TiO_2 . Previous research showed that silver bromide (AgBr) with a band gap of 2.6 eV is an inorganic photosensitive semiconductor material, which will have high photographic sensitivity under visible light irradiation and can be used to modify TiO_2 to have visible light activity [15].

Hu et al. prepared Ag/AgBr/ TiO_2 nanocomposites by the deposition-precipitation method. The catalyst showed high visible light photoexcited efficiency when investigated for the decomposition of azodyes and killing of *Escherichia coli* [16]. Zang and Farnood synthesized the photocatalysts with several levels of AgBr contents on TiO_2 also by the deposition-precipitation method. In the investigation of methyl orange's (MO's) degradation, they found that at higher levels of AgBr content (>9%) the degradation of MO was dependent on the incident light intensity, while at lower contents the degradation rate increased with the increase of incident light intensity and finally reached a plateau level [17]. Similarly, Liu et al. prepared a series of AgBr/ TiO_2 visible photocatalysts with different mass ratio of $m_{\text{AgNO}_3}/m_{\text{TiO}_2}$ using $\text{Ti}(\text{OC}_4\text{H}_9)_4$, KBr, and AgNO_3 as precursors. The result showed that the homogeneously dispersed coupled heterojunction microstructures were formed while $m_{\text{AgNO}_3}/m_{\text{TiO}_2} = 3.35$ which have stronger absorption in the whole visible region and are beneficial to the further improvement of photocatalysis [18].

Ag-AgBr/ TiO_2 composites prepared by a sol-gel method followed by photoreduction showed intrinsic antibacterial activities against *Escherichia coli* in the dark. Under visible light irradiation, inactivation of *E. coli* over these Ag-AgBr/ TiO_2 composites was attributed to both their photocatalytic disinfection activities and intrinsic antibacterial properties [19]. Wang et al. synthesized Ag/AgBr/ TiO_2 via sol-gel (Ag/AgBr/ TiO_2) route and solvothermal route (S-Ag/AgBr/ TiO_2), respectively. The test of ibuprofen degradation and mineralization revealed that Ag/AgBr/ TiO_2 's photocatalysis is a little better than S-Ag/AgBr/ TiO_2 , and they both were much superior to the single-component (TiO_2) and two-component (Ag/ TiO_2 , Ag/AgBr) systems [20]. The charge transfer mechanisms in the composites have not been revealed clearly, so Wang et al. developed a simple microemulsion-like chemical precipitation method to construct AgBr/ TiO_2 composite. The most difference of the method is the dual role of Br^- in the synthetic process, as linkers between cetyltrimethyl ammonium cation surfactants and nanocrystalline anatase TiO_2 in the acidic condition

FIGURE 1: The mechanism of AgBr/TiO₂.

and as bromine sources to directly produce nanocrystalline AgBr on the surfaces of TiO₂ by chemical precipitation. This work found that the Br⁻ in crystal could effectively capture photogenerated holes, consumedly favoring charge separation, whose factors are responsible for the high activity and excellent stability of the AgBr-TiO₂ [21].

Graphene is a single layer which has many outstanding properties, such as high theoretical specific surface area, superior mechanical, chemical stabilities, and so forth. Most recently, Wang et al. synthesized a new photocatalytic nanocomposite, Ag-AgBr/TiO₂ supported on reduced graphene oxide (Ag-AgBr/TiO₂/RGO). The four-component nanocomposite (Ag-AgBr/TiO₂/RGO) exhibited a much higher photocatalytic activity for the degradation of penicillin G (PG) under white light-emitting diode (LED-W) irradiation, compared with the single-component (TiO₂), two-component (Ag-AgBr, Ag/TiO₂, and TiO₂/RGO), and three-component (Ag-AgBr/RGO, Ag-AgBr/TiO₂ and Ag/TiO₂/RGO) nanocomposites [22].

To well understand the properties of photogenerated charges and their effects on the photocatalytic performance, a detailed mechanism schematic of transfer and separation of photogenerated charges is presented, as shown in the Figure 1. In the schematic, the energy levels of conduction band (CB) bottom and valence band (VB) top of AgBr and TiO₂, along with the affinity level of O₂, are carefully labeled, which are important to reasonably reveal the transfer and separation processes of photogenerated charges. According to [23], the E_{VB} levels for TiO₂ and AgBr stand at 2.86 eV and 1.50 eV via the standard H₂ electrode (SHE), respectively, while their ECB levels are positioned at -0.32 eV and -1.06 eV.

Moreover, the valence band potential of a semiconductor at the point of zero charge also can be calculated by the following empirical equation $E_{VB} = X - Ee + 0.5Eg$ [24], in which E_{VB} is the VB top potential via the SHE, and the CB bottom potential can be determined by $E_{CB} = E_{VB} - Eg$. Thus, the E_{VB} levels for TiO₂ and AgBr stand at 2.09 eV and 0.42 eV, respectively, while their ECB levels are positioned at -1.09 eV and -2.14 eV. Based on the levels of E_{VB} and E_{CB} by calculating and obtaining from the references, it is confirmed that the levels of E_{VB} and E_{CB} of AgBr are higher than those of TiO₂. In general, the O₂ affinity level (O₂/O₂^{•-}) stands at about 0 eV (-0.046 eV versus SHE) [25]. Thus, it is assumed that the photogenerated electrons at the E_{CB} of TiO₂ and AgBr would be effectively captured by the adsorbed O₂, leading to the SPS occurrence at the DRS edge.

In order to investigate the mechanism of AgBr/TiO₂, Velmurugan and Swaminathan [26] use the PL emission spectra to analyze the samples. It can be discovered that the positions of the peaks are similar, when comparing AgBr/TiO₂ with pure TiO₂. However, the PL intensities of TiO₂ are higher than AgBr/TiO₂. The phenomenon of the reduction emission intensity of AgBr/TiO₂ implies that the recombination of charge carriers is effectively inhibited by AgBr nanoparticles. In addition, it also can be proved by the photocatalytic activity test. From the test, it can be found that in the presence of AgBr/TiO₂ the degradation of RR 120 reached 98% in 40 min under solar light, while 60.1% was observed with TiO₂.

In order to promote the photocatalytic activity or stability, an appropriate synthesis approach and the ration of the different component are all the key aspects. Consequently, optimization of these two matters in the final results. And if

there is a selected substrate to support the material, it will be more efficient under irradiation.

2.3. AgI/TiO₂. As a member of the silver halides, silver iodide (AgI) has three different phases: at room temperature, it is usually a mixture of a hexagonal β -AgI phase and a cubic γ -AgI phase; above 147°C, β -AgI undergoes a first order phase transition into the α -AgI phase. When at ambient conditions, the AgI with two distinct phases has a direct band gap of about 2.9 eV [27]. Hu et al. prepared photocatalyst Ag/TiO₂ by the deposition-precipitation method that showed high effective in killing *Escherichia coli* and *Staphylococcus aureus*. The Ag/TiO₂ also revealed that the photocatalytic degradation of the cell structure caused the cell death by TEM, potassium ion leakage, lipid peroxidation, and FT-IR measurements [28]. Li et al. synthesized a core/shell/shell nanostructured AgI/Ag-I₂/TiO₂ by a feasible approach with AgNO₃, LiI, and Ti(Obu)₄. The photocatalytic tests show that the prepared photocatalyst exhibited 4 and 6 times higher than P25 supported AgI, when used to degrade crystal violet and 4-chlorophenol [29]. Wang and Liu synthesized Ag/AgI supported TiO₂ acid corrosion nanobelts plasma photocatalyst. The test of degradation of methyl orange (MO) showed the enhancement of the visible light activity, because of the larger area of the photocatalyst after acid corrosion [30]. Shila's investigation indicates that modified TiO₂ with AgI also enhanced the adsorption of methyl violet. What is more, they showed the modification of TiO₂ nanoparticles with AgI has no effect on the rate of adsorption but it results in a stable adsorption and higher adsorption capacity. The kinetic test reveals that methyl violet (MV) adsorption onto AgI/TiO₂ followed pseudo-first-order model; however, adsorption of MV onto TiO₂ followed simplified Langmuir model [31]. Since direct precipitation of AgI on the supports of TiO₂ would limit the interfacial charge transfer, a three-dimensional nanostructure with irregular shapes and unclear interfaces has been developed. An et al. fabricate a 3D AgI/TiO₂ nanophotocatalyst with an acanthosphere-like morphology composed of nanothorns, which has enhanced performance towards degradation of organic contaminants, in comparison with the conventional nanoparticles and nanotubes [32].

It has been reported that the inner electric field of heterojunction catalyst can decrease the recombination of the electron-hole pairs and prompt the photocatalytic activity of semiconductor photocatalysts [33]. And composite particles with different AgX heterojunction with better photographic properties than single AgX have been studied in the field of photographic films [34].

Cao et al. prepared a composite catalyst AgI/AgCl/TiO₂ by ion exchange method. And the result indicates that when the molar percentage of AgI to initial AgCl is 20%, the composite catalyst has the maximal degradation efficiency of MO [35].

Specialized morphology of the particles may lead to specific properties, so the 3D nanoparticles with high specific surface area have an enhanced ability of photocatalysis. And multiselectronics' common effect also may enhance the initial properties of the single material.

3. Conclusion and Outlook

AgX is instable when exposed to sunlight alone. And TiO₂ shows relative activity under UV light. However, AgX/TiO₂ system showed high photocatalytic activity and stability under visible light. Great efforts have been made to develop AgX/TiO₂ preparation methods in recent years. In this review, AgX/TiO₂ composite semiconductors with different morphologies, such as Ag/AgX composite, nanotube or other nanostructures, thin film fabrication, and reduced graphene oxide supported AgX/TiO₂, were prepared to enhance their visible light activity and circulation stability through hydrothermal, ion-exchange methods or precipitation processes.

It was assumed that properties, such as stability, electron-hole recombination rate, and visible light photocatalytic activity, of AgX/TiO₂ strongly depended on the preparation method used. However, the properties of AgX/TiO₂ cannot be improved by a single preparation method. Therefore, a major area of future research would be the development of AgX/TiO₂ preparation method, such as preparation of TiO₂ coupled with various types of AgX, ternary composite of AgX/TiO₂ and other semiconductors, and AgX/TiO₂ loaded on supported materials. Such materials preparation together with the development of technically application is crucial for broader scale utilization of photocatalytic systems in commercial application.

Conflict of Interests

The authors declare that there is no conflict of interests regarding the publication of this paper.

Authors' Contribution

Xiaojia Wan and Ting Wang contributed equally to this work and share first authorship.

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