Chapter 7
Surface Plasmon-Based Nanomaterials as Photocatalyst

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Contents

Abstract In recent era, plasmonic photocatalysts have facilitated rapid progress in improving the photocatalytic efficiency under visible light irradiation, increasing the prospect of using sunlight for environmental and energy applications, such as wastewater treatment, water splitting, and carbon dioxide reduction. Plasmonic photocatalysis makes use of noble metal NPs dispersed in semiconductor photocatalysts and has two prominent features, a Schottky junction and localized SPR effect. With the advances in fundamental and experimental studies on plasmon-mediated photocatalysis, the rational design and synthesis of metal/semiconductor and carbon-based hybrid nanostructures as photocatalysts have been realized. This chapter highlights a recently reported and easy methodology for the fabrication of SPR-based materials and its real developments in plasmon-mediated photocatalytic mechanisms, such as Schottky junctions, direct electron transfer, enhanced local electric field, plasmon resonant energy transfer, and scattering and heating effects. In addition, this chapter also summarizes the factors, size, shape, geometry, loading, and composition of plasmonic metal, as well as the nanostructure and properties of

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semiconductors that mainly affect the photodegradation of dyes. Finally, a perspective on future directions within this rich field of research is provided.

Keywords Metal nanoparticles · Au and Ag · Surface plasmon resonance · Visible light · Photocatalysis · Water treatment

7.1 Introduction

Visible light sources are a clean, renewable, and abundant energy alternative to fossil fuels (Liu et al. 2011). In addition to direct light-to-electricity conversion, photocatalysis provides an alternative method for storing energy in chemical bonds that can be released later without producing harmful by-products (Zeitler 2009). This is a kind of green chemical synthesis because of its abundance and environment-friendly nature. Considerable efforts have been made to use visible light as a driving force for chemical synthesis processes. Nevertheless, the potential applications of photochemical reactions have been limited by the inability of most organic molecules to absorb light in the visible range of the spectrum (Lang et al. 2014). Consequently, recent reviews on general organic photocatalysis highlighted the design of highly efficient photocatalysts to bring significant progress in an easy, scalable, and biogenic synthesis (Lang et al. 2014; Daniel and Astruc 2004; Wang et al. 2012a). Plasmonic photocatalysis has recently come into focus as a very promising technology for the high-performance photodegradation of harmful dyes (Wang et al. 2012a; Nan Zhang et al. 2012; Linic et al. 2011). This process involves the dispersal of noble metal nanoparticles (mostly Au and Ag, tens to hundreds of nanometers in size) into semiconductor photocatalysts and obtain drastic enhancement of photoreactivity under the visible light irradiation (Linic et al. 2011; Yu et al. 2011; Nan Zhang et al. 2014). Extensive work has been made to develop effective photocatalysts for the degradation of organic harmful pollutants (Ahmad et al. 2015). Dyes are generally toxic, carcinogenic, and harmful with adverse effects on human and animal health (Nan Zhang et al. 2012; Jian Long Wang and Xu 2012). Most dyes are recycled because their release into the aquatic and marine environments could be a source of severe ecological pollution (Chong et al. 2010; Yu et al. 2010). Different kinds of dyes have many applications in numerous industries, including, plastic, paper, rubber, furniture, textile, concrete, and medicine (Yu et al. 2010). Among the industries, the textile industry is the main consumer of toxic dyes. Approximately 10% of the dyes used in industry are liquidated directly into the atmosphere as a pollutant, which is ecologically unsafe and esthetically unacceptable (Lettmann et al. 2001). Based on several studies, SPR-based nanomaterials have promising applications in dye degradation for wastewater treatment. At present, the most extensively studied nanomaterials for wastewater treatment mainly include zerovalent metal nanoparticles/nanostructures and carbon-based metal nanostructures. (Mingkui Wang et al. 2010).

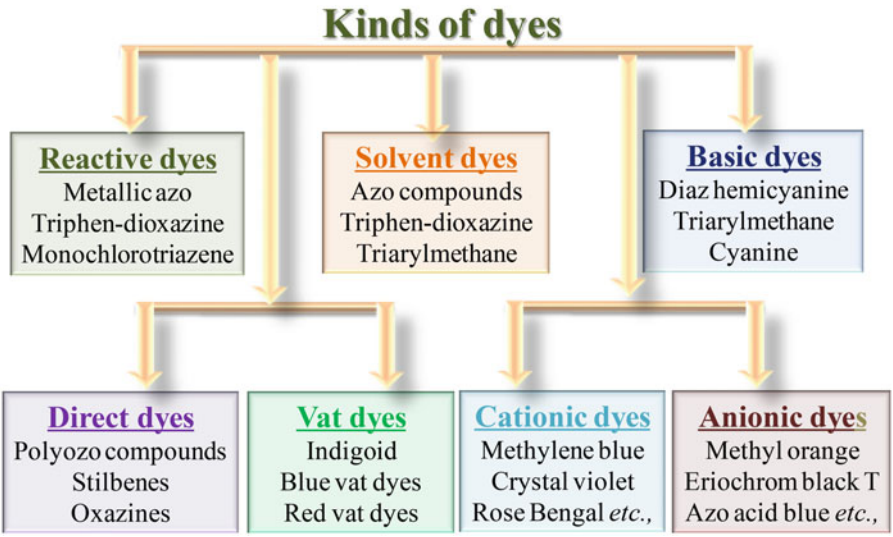

Fig. 7.1 Different category of dyes and its derivatives

Figure 7.1 represents the different categories of dyes that are openly released as waste in aqueous streams from several industries (Camarero et al. 2003; William IV et al. 2008). The direct release of enormous amounts of toxic dyes is unavoidable because the individual textile industry consumes immense amounts of water and dyes are not consumed completely by the fibers during the dyeing procedure (Muhd Julkapli et al. 2014; Jie et al. 2013). These high concentrations of dyes in effluents interfere with the dispersion of visible light in water, causing interference to photosynthesis and a decrease in gas solubility. Furthermore, artificial dyes, which contain an aromatic ring in their structure, are noxious and carcinogenic compounds (Salleh et al. 2011; He et al. 2011). Photocatalytic reactions can be classified into two types: homogeneous and heterogeneous photocatalysis. The most prominent features of the photocatalytic system are the required band gap, suitable morphology, high surface area, stability, reusability, and special SPR effect. This chapter focuses on the recent progress in the fabrication, decoration/anchoring, modification, and water treatment applications of SPR-based effect of metal NPs as photocatalysts, which provides perspectives on future water treatment developments.

7.2 Metal (Au and Ag) Nanoparticles

Noble metals, such as gold (Au), silver (Ag), platinum (Pt), and palladium (Pd), have been assessed for potential visible light applications. Particular attention on Au and AgNPs has been studied widely because of their unique optical and electronic properties together with their many applications in electronics, photonics, catalysis,

and nano-biotechnology (Daniel and Astruc 2004; Burda et al. 2005). AuNPs have also attracted remarkable attention for both heterogeneous and homogeneous visible light-induced catalysis (Haruta 2005). Recently, the localized surface plasmon resonance (LSPR) effect of nanostructured materials, e.g., Ag and AuNPs, has been applied successfully to photocatalysis under visible light irradiation and shown to be quite promising. Therefore, the present chapter deals selectively with plasmonic nanogold photocatalysis with the main focus on the following (Hashmi and Hutchings 2006):

(i) Basic concepts of the SPR effect of metal NPs.
(ii) Metal NP-based plasmonic photocatalyst preparation methods.
(iii) Recent developments in the efficient Au- and AgNP-based plasmonic photocatalysis.
(iv) Application of plasmonic photocatalysis to energy conversion processes that are driven by visible light.
(v) Possible reaction mechanisms of SPR-enhanced photocatalytic activity: In conclusion, the challenges and probable future applications will be highlighted.

7.3 Surface Plasmon Resonance Effect

Mie's theory defines the special SPR as the resonant photon-induced coherent oscillation of charges at the metal–dielectric interface that are recognized when the photon frequency matches the natural frequency of the metal surface electrons oscillating against the restoring force of their positive nuclei (Sarina et al. 2013).

When the wavelength of incident light is in the range of the SPR absorption of noble metal NPs, the electrons are excited by SPR to the conduction band (Fuku et al. 2013). For example, the resonance energy of AuNPs usually occurs in the visible range ($\lambda = 530$ nm, depending on the size, shape, and diffraction index of the medium of AuNPs). The interaction between the resonant photons and the surface electrons results in a high absorption coefficient of photons in resonance with the plasmon excitation and capacitive coupling between clusters of plasmonic AuNPs. Figure 7.2 presents the process of the displacement of electron density. The columbic restoring force that is caused by oscillations in the NP is established, and the resonance between the oscillations and incident light is known as SPR (Kelly et al. 2003).

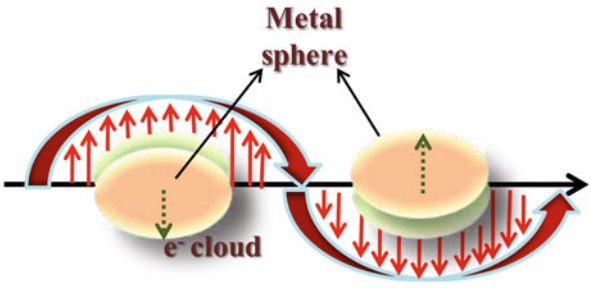

Fig. 7.2 Schematic diagram of SPR in small spherical metallic NPs under visible light irradiation

Mayer et al. described the basis of the SPR effect in a review (Link and El-Sayed 2000) and provided detailed illustrations of the mechanisms of the plasmonic enhancement to photocatalysis. This review provided a better understanding of the physical principles of the special effect of SPR and hence with only brief introduction. When small spherical metallic NPs are under visible light irradiation, the number of electrons decreases on one side of the NPs and increases on the other side. This causes a redistribution of charge density. The redistributed charge density produces an electric field inside and outside the metal NPs with a direction that is opposite to that of the electric field of the light, as shown clearly in Fig. 7.1.

7.4 Basic Concepts and Beneficial Effects of SPR

The basic concept of special SPR effect is comprised of the following. A collective oscillation of free electrons in metal NPs is driven by the electromagnetic field of incident light, in which the metallic NPs absorb visible and infrared light in particular regions. For example, Au and AgNPs show a strong photoabsorption band of visible light because of their surface plasmon, which displays maxima at approximately $\lambda = 530$ and $\lambda = 400$ nm, respectively (Khan et al. 2015a, b, c). On the other hand, nanostructured AgNPs are oxidized easily, whereas AuNPs are more chemically stable in the presence of oxygen (Rayalu et al. 2013).

Recently, we successfully introduced this unique property to the new field of plasmonic photocatalysts to catalyze organic reactions and water splitting (Khan et al. 2015a, b, c). Plasmonic photocatalysis offers a new opportunity to solve the limited efficiency of photocatalysts and photovoltaic devices. In these reactions, nanostructured plasmonic metals are combined frequently with a semiconductor-based material (e.g., SnO_2, TiO_2, WO_3, and CeO_2, namely, plasmonic photocatalysts), and the catalytic activity and efficiency are enhanced greatly by the SPR effect that improves the solar-energy-conversion efficiency in the following aspects (Khan et al. 2015a, b, c, 2016; Nayak et al. 2017):

(i) Near-field enhancement of localized plasmon
(ii) Increase of the scattering effect
(iii) Excitation of e^-/h^+ pairs in the semiconductor due to the plasmonic energy transfer from the metal to the semiconductor

Figure 7.3 shows the special beneficial effects of plasmonic catalyst under visible light irradiation. Plasmonic photocatalysis has attracted recent attention as a hopeful technology for the high-performance photocatalytic degradation of harmful dyes (Kochuveedu et al. 2013; Wang et al. 2012a), (Nan Zhang et al. 2012; Linic et al. 2011). This involves the distribution of noble metal nanoparticles (generally Au and AgNPs) into semiconductor photocatalysts and obtains strong enhancement of the photoreactivity under a broad range of visible light. The use of noble metal nanoparticles has numerous benefits in the field of photocatalysis.

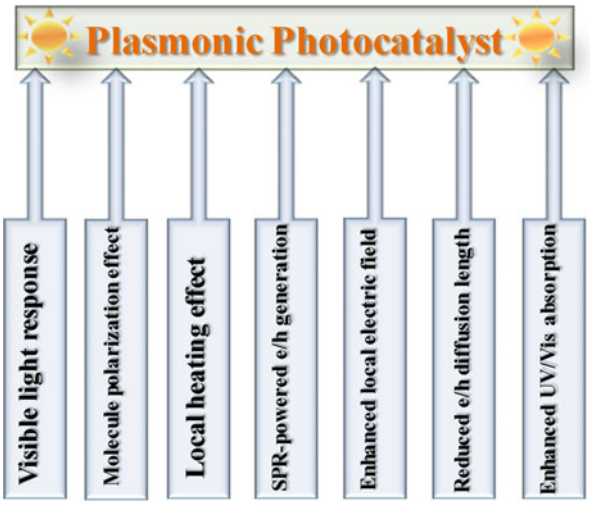

Fig. 7.3 Major special beneficial effects of SPR for improved photocatalysis

Compared to the common semiconductor photocatalysis, plasmonic photocatalysis has two distinct features: Schottky junction and localized surface plasmon resonance; each feature benefits photocatalysis in a different way. For example, a Schottky junction results from the contact of a noble metal and semiconductor. This builds up an internal electric field in a region inside the photocatalyst but close to the metal/semiconductor interface. This would force the electrons and holes to move in different directions once they are formed inside or near the Schottky junction (Wang et al. 2012b). In addition, the metal fraction provides a quick lane for charge transfer, and its surface acts as a charge-trapping center to host more active sites for photoreactions. The Schottky junction and the fast lane charge transfer work together to reduce electron–hole recombination charge (Chang et al. 2007; Xuming Zhang et al. 2013).

7.5 Preparation Methods of Metal NPs as Photocatalysts

Various sets of chemical, physical, and biological methods for the preparation of plasmonic photocatalysts are available. Examples include chemical reduction, physical vapor deposition, hydrothermal, and electrochemically active biofilm-assisted synthesis methods for the fabrication of plasmonic photocatalysts (Zhu et al. 2010; Hou and Cronin 2013; Wang et al. 2012a; Khan et al. 2015a, b, c). A series of synthesis methods have been adopted for the fabrication of metal NPs that would be ideally controllable in terms of their shape, size, morphology, cost, environmental friendliness, and high product yield with fewer waste products (Fig. 7.4).

Metal nanoparticles show a high abundance of surface plasmon excitation. For example, Au and AgNPs have attracted considerable attention because of the spatial

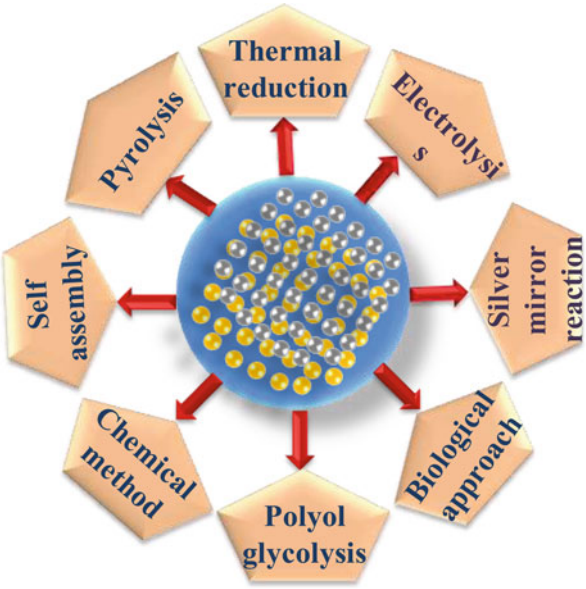

Fig. 7.4 Several preparation methods for the metal nanoparticles

SPR effect in the visible spectrum range, which can be used in numerous environmental remediation applications (Sun and Xia 2002; Jian Zhang et al. 2009). The surface plasmon properties of metal NPs are dependent on their shape, size, and morphology. The specific morphology of metal NPs is a noteworthy concern that affects their special plasmonic behavior (Gramotnev and Bozhevolnyi 2010). Several methods have been used for the fabrication of metal NPs with a controlled size and shape. The metal precursor is mixed with a reducing agent in the presence of a stabilizing agent that is used to control the size and shape of metal NPs. Similarly, $AgNO_3$ (silver nitrate), as a precursor, can be used because of its low cost and high abundance. More than a few reducing agents, such as sodium citrate, sodium borohydride, and electrochemically developed biofilms are used to reduce the metal ions present in solution to metal, metal oxide/Ag nanocomposites. On the other hand, in most cases, stabilizing/capping agents are introduced to manage and stabilize the morphology of the resulting metal NPs. Despite this, in photochemical synthesis, a variation of light treatment methods is assumed to produce metal NPs. Light-mediated synthesis has been applied to the fabrication of NPs, e.g., laser ablation or direct laser irradiation of an aqueous solution of metal salt precursor in the presence of a surfactant to make an accurate shape, size, and distribution of metal NPs, where the light source works as a reducing agent (Yee et al. 1999; Lim et al. 2006; Sharma et al. 2009).

The biological route is considered a green and sustainable methodology that has attracted substantial attention because of its potential to address the energy and environmentally related issues. The development/growth of biofilms using microorganisms is a biological approach for the fabrication of different kinds of metal nanoparticles and nanocomposites (Kalathil et al. 2011). Bio-electrochemical

systems (BESs) use microorganisms as a catalyst in electrochemical reactions. The best recognized BES is the microbial fuel cell (MFC), which can be used in metal-consuming microorganisms to transform the chemical energy of the substrates dissolved in wastewater to electrical energy. BESs can be used for power generation from wastewater, power-driven electricity production, bioremediation, and biosensing applications. In recent times, mixed culture-developed biofilms were reported to be a biogenic reducing tool for the synthesis of NPs (Au, Ag, and Cys − Ag) and metal-graphene nanocomposites (Khan et al. 2012, 2013, 2015a, b, c). The main benefit of these procedures is that the mixed culture-developed biofilms are used as the reducing tool that does not involve an exterior energy contribution, toxic chemicals, or expensive solvents. In addition, the reactions occur at room temperature, which makes the formation of nanoparticles/nanocomposites highly efficient.

7.6 Effect of Plasmonic Resonance in Photocatalysis

7.6.1 Plasmonic Resonance Mechanisms

In highly conductive nanocomposites, free electrons are confined locally. When the as-fabricated nanomaterials are irradiated with electromagnetic energy at the plasma frequency, the spatial electron density redistributes and produces an electric field. Concurrently, a columbic restoring force of the positively charged surface nuclei is present and induces collective oscillations of the charges in the particle, which are similar to an oscillating spring after stretch and release (Warren and Thimsen 2012). Such oscillations of electrons and electromagnetic fields are defined as localized surface plasmons. In the state of localized surface plasmonic resonance (LSPR) induced by the radiation of a specific LSPR wavelength, the free electrons will oscillate with the maximum amplitude. LSPR is characterized by a buildup of intense, spatially nonhomogeneous oscillating electrical fields in the vicinity of the nanostructure (Linic et al. 2011). In such a way, the energy of incident radiation is transferred to the plasmonic particles. The LSPR profile can be tuned by tailoring several parameters, such as their nano-size, shape, interparticle distance, and nature of the surrounding medium (Linic et al. 2011; Clavero 2014; Liz-Marzán 2006; Kale et al. 2013).

The fundamental principle in the photocatalytic degradation of dyes or pollutants is when the metal nanoparticles, as a catalyst, are irradiated with photons with an energy greater than or equal to their band-gap energy (E_g), an electron (e^--cb) is excited from the valence band to the conduction band, which leaves a hole (h^+-vb). The excited electrons and holes then migrate to the surface of another state. The rate of charge recombination is inhibited frequently by a scavenger or another doped material, which can easily trap the electrons or holes. Accordingly, better crystalline nanostructure materials with fewer defects can minimize the trapping states and charge recombination sites, which results in improved efficiency in the use of the

photogenerated transporters for the desired photoreactions. For higher photocatalytic efficiency, the electron–hole pairs should be separated well, and the charges should be transferred rapidly across the surface/interface to impede recombination (Linic et al. 2015).

7.7 Photocatalytic Dye Decomposition Processes

Photocatalysis can be used for the degradation of environmentally harmful dyes, photocatalytic hydrogen evolution, and photosynthesis of useful chemicals. The basic mechanisms of nano-semiconductor-based photocatalysis comprise photochemical processes of visible light absorption, electron-hole pair generation, separation, and free charge carrier-induced redox reactions. This is beneficial for a wide range of applications, such as wastewater treatment, air purification, water splitting, and the self-cleaning of surfaces (Linic et al. 2015).

Table 7.1 lists the performances of novel plasmonic nanocomposites with graphene sheets as base materials. Numerous studies have chosen organic dyes as the pollutant because the degradation process can be examined simply through the variations in the photoabsorption of the responding or degrading solution.

Table 7.1 Comparative results of photocatalytic dye degradation performances using different nanostructures

Nanocomposites	Dye as pollutant	Light source	Degradation result	References
Au-graphene	Methylene blue	($\lambda > 420$ nm)	64%	Khan et al.
Ag-graphene	Methylene blue and Congo red	($\lambda > 420$ nm)	65% and 90%	Khan et al.
Pt-graphene/TiO_2	Methylene blue	(8 W, $\lambda > 420$ nm)	93%	S.yYe et al.
Graphene-gold	Methylene blue, RhB and orange II	($\lambda > 420$ nm)	88.6%, 27.6%, and 8.5%	Zhao et al.
Pt-Pd-graphene	Basic fuchsin and indigo carmine dyes	($\lambda > 420$ nm)	70% and 65%	Kurt et al.
Pt/graphene	Rhodamine B and methylene blue	8 W, halogen lamp	70% and 82%	Oh et al.
Au@TiO_2-graphene	Acid blue 92	125 W mercury lamp	72% and 43%	Setayesh et al.
		($\lambda > 574$ nm)		
Au@TiO_2/graphene	2,4-dichlorophenol (2,4-DCP)	($\lambda > 420$ nm)	78%	Shi et al.
Ag-Au on graphene sheets	4-nitrophenol	($\lambda > 420$ nm)	97.38%	Dhole et al.
Au–Pd-reduced graphene oxide	2-chlorophenol	Sunlight	100%	Yamauchi et al.

7.7.1 Photochemical Mechanism

Photocatalysis can be applied effectively to the photodegradation of environmentally harmful matter, photocatalytic hydrogen evolution, and photosynthesis of useful chemicals. The emerging technologies have attracted considerable research interest. The basic mechanisms of nano-semiconductor-based photocatalysis involve the photochemical processes of light absorption, electron-hole pair generation, and the separation and free charge carrier-induced redox reactions, as shown in Fig. 7.5.

Metal/semiconductor hybrid structures are usually employed for plasmonic photocatalysis applications. There are some insightful review addressing the mechanisms (Hou and Cronin 2013; Jiang et al. 2014). The charge-transfer mechanism also affects the photochemical selectivity of LSPR-mediated photocatalysis (Xuming Zhang et al. 2013). In a study of the SPR-mediated oxidation of p-aminothiophenol (PATP), when Au nanoparticles were used as a catalyst, the LSPR-mediated oxidation of PATP yielded p,p-dimercaptobenzene (DMAB). On the other hand, no DMAB evolution was detected when the AuNPs were loaded onto a metal oxide (TiO_2 NPs) under LSPR spectral irradiation, as the hot electrons were injected from the AuNPs to the conduction band (CB) of TiO_2 NPs. When UV illumination was introduced, p-nitrophenol (PNTP) formed from PATP in a single step, which was attributed to electron transfer from the UV-excited TiO_2 to AuNPs. Interestingly, when the UV illumination was stopped, the PNTP molecules were reduced further to DMAB. This suggests that the charge-transfer mechanism may play an important role in LSPR-mediated photocatalysis to manipulate the reaction activity, product formation, and selectivity (Hou and Cronin 2013; Jiang et al. 2014).

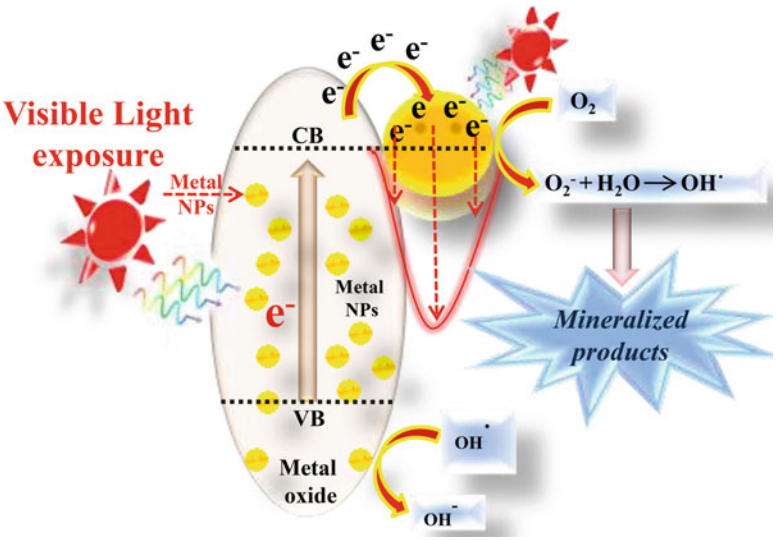

Fig. 7.5 Schematic diagram of plasmonic-mediated photocatalysis under visible light irradiation

The photodegradation of organic pollutants by semiconductor-based photocatalysts is an economical, clean, and effective means for water and air purification. In typical aqueous solution-based photocatalytic reactions, upon photoexcitation, either the photogenerated energetic charge carriers will react with the solvent to form highly reactive radicals that can break the chemical bonds of the organic pollutants or the charge carriers will be transferred directly to the reactants adsorbed on the surface of the catalysts to induce photochemical transformations. Either way, the organic pollutants are degraded photocatalytically into short-chain molecules or eventually into CO_2 and H_2O. The photocatalytic reactions are strongly dependent on the absolute surface area, light absorption properties, energy band level, and charge separation potential of the photocatalyst. The beneficial surface plasmonic effect on photocatalysis has been proven. Upon plasmonic excitation, the elevated field increases the rate of energetic charge carrier generation, resulting in a higher probability of redox reactions (Ni et al. 2007; Priebe et al. 2015). Efforts have been made for better morphology control, more suitable energy coupling, and novel hybrid structures to achieve higher efficiency and better stability of the catalyst. Many studies chose organic dyes as the representative pollutant because the degradation process can be monitored simply through changes in the photoabsorption of the reacting solution.

7.8 Conclusion

This study reviewed the recent developments of plasmon-enhanced photocatalysis, particularly on the three major functions of the plasmonic effect, i.e., (i) light absorption, (ii) hot electron injection, and (iii) near-field enhancement. The plasmon-based mechanisms and their photonic effects showed that the plasmon effect is a very promising strategy to improve the photocatalytic performance of metal/metal oxides. Significant advances have been made to extend the technology for practical applications. In the future, constant research should address the following concerns:

1. Although the plasmonic enhancement has been found to be useful for photocatalysis applications, considerable effort is needed to develop the material and architecture for optimization.
2. The photostability and chemical stability of plasmonic materials are not up to the work particularly when complicated morphologies with high structural energies are required. While many studies have reported more robustness of the plasmonic materials, the exchange between protection and efficiency needs to be addressed.
3. Bare Au and AgNPs are effective but expensive. Low-cost plasmonic metals with carbon-based materials, such as Ag-graphene/*g*-C_3N_4 or Au-graphene/*g*-C_3N_4, should be fabricated using easy and scalable methodologies.

4. The limited understanding of the interaction between plasmonic materials and electrolyte in plasmonic effects hinders the development of novel, effective materials.

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