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Green Synthesis of Nano-Semiconductor Photocatalysts and their Effectivity for Photo-Oxidation of Organic Pollutants in Wastewater: A Review Study

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Review Article

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Abstract

In recent years, green synthesis of nanoparticles (NPs) has been regarded as preferable and acceptable than chemical approaches, particularly when it applies to pollution control and wastewater treatment. The green synthesis approaches were effectively employed to prepare a huge number of semiconductors photocatalysts, such as TiO_2, WO_3, SnO_2, CeO_2, ZnO, Bi_2O_3, Ag, Na_2SeO_3, $ZnCr_2O_4$, Nb_2O_5, and so on. Photocatalytic degradation is an advanced water treatment technology that has great potential for the photo-oxidation of various organic pollutants and various types of nano-semiconductors photocatalysts. Plant waste has the potential to be more useful in the green synthesis of metal nanoparticles (NPs) due to their availability, high biodiversity, eco-friendliness, and low cost. This review focuses on the employing of plant extracts in the synthesis of nano-photocatalysts as a cost-effective approach. Our study involves reviewing a huge number of impactful studies that deal with the photooxidation of organic pollutants under light radiation. It was concluded that the utilization of waste plants in the green synthesis of nano-photocatalysts is a low-cost technology, environmentally friendly, and can produce nanoparticles with outstanding efficiency for water pollution remediation. This study encourages the development of new technologies based on reusing plant waste in the synthesis of economic photocatalysts for environmental applications.

Keywords: Green Synthesis F Nps; Semiconductor Photocatalysis; Organic Oxidation; Wastewater Treatment

Abbreviations: AOP: Advanced Oxidation Processes; HOP: Hazardous Organic Pollutants; NP: Nanoparticles; EBT: Eriochrome Black T.

Introduction

A few days ago, organic pollutants revealed a greater threat to human health and the environment due to the potential contamination of marine life and ecosystems via phenols, antibiotics, pesticides, and dyes [1,2]. These pollutants exhibit a strong toxic effect and are difficult to decompose in nature [3-5]. Thus, it is crucial to develop robust technology to effectively eradicate organic contaminants. As a prominent solution, advanced oxidation processes (AOPs) are a set of efficient chemical treatment techniques that can completely oxidize recalcitrant organic materials

in wastewater into simple products [6]. AOPs (see Fig. 1) involve the production of powerful and non-selective radical species that can break down refractory organic pollutants in efficient degradation pathways [7-10].

Nowadays, a huge number of hazardous organic pollutants (HOPs), like organic dyes [11,12], pesticides [13], phenols [14], antibiotics [14-16], detergents [17], drugs [18], biphenyls [19], plasticizers [20], and others, are discharged into water and treated by efficient treatment technologies. Among them, heterogeneous photocatalytic degradation technology is an advanced oxidation process (AOPs), which has received more attention due to its advantages, such as low energy consumption, mild reaction conditions, and a broad range of applications [21,22]. Under light illumination, it has been shown to be more efficient in degrading HOPs into low-toxic and biodegradable products, which can then be completely mineralized into CO_2 and H_2O [23,24] (Figure 1).

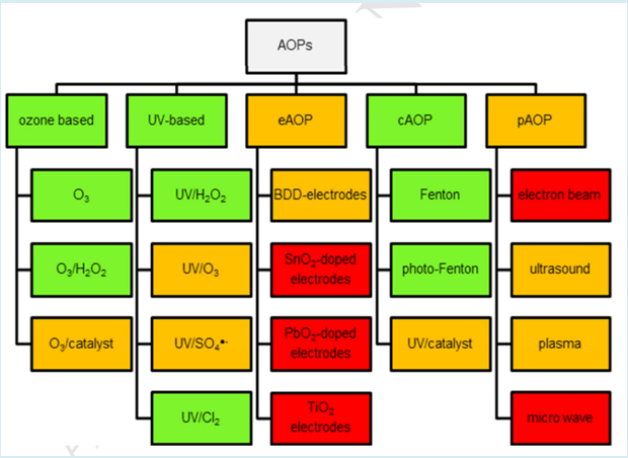

Figure 1: Classification of AOPs that can be employed for oxidation of different organic pollutants [25].

A huge number of metal oxides, such as TiO_2, WO_3, SnO_2, CeO_2, ZnO, Bi_2O_3, Ag-based photocatalysts, Nb_2O_5, and so on, were employed as semiconductor photocatalysts for the degradation of organic pollutants under UV and visible light sources [26-28]. Several techniques and approaches have been implemented to synthesize the metal oxides, including sol gel [29], liquid phase [30], chemical vapor deposition [31], colloidal [32,33], electrodeposition [34], hydrothermal method, co-precipitation, solvothermal, self-assembly, and so on [35]. The green synthesis technologies based on bio-resources have been proven to be more effective, environmentally friendly, and less dangerous than other methods [36-39]. These methods were developed to produce a variety of metal oxide nanoparticles derived from biocompatible materials such as microorganisms (bacteria, algae, and fungi) and plant extracts (steam, seed, leaf, fruit, and flower) [40,41].

Recently, the synthesis of nanoparticles from plant extracts has received increased attention due to their activity and economics. Hence, many plant species were investigated for the preparation of nanoparticles in various shapes and sizes. The researchers gave priority to fabricating plant-mediated NPs because dealing with plant parts is easier and more practical than dealing with microorganisms (bacteria, algae, and fungi). Moreover, the utilized microorganisms require a significant amount of time to cultivate fungus or bacterial cultures to be ready for practical use. On the other hand, plant-mediated NPs exhibited many other advantages, like their availability, high biodiversity, eco-friendliness, and low cost [42]. This review highlights the recent technologies employed to fabricate metal-oxide nanoparticles using different plant parts. Moreover, this study concentrates on the application of green synthesized NPs in the photocatalytic oxidation of organic pollutants in wastewater. This study encourages the development of new technologies based on reusing plant waste in the synthesis of economic photocatalysts for environmental applications.

Fundamentals of Photocatalytic Reactions

The photocatalysis process "has been proven to be promising and sustainable for addressing the problems related to environmental pollution and the energy crisis by harvesting clean or renewable solar energy [43]. The photocatalysis process was commonly implemented to degrade the organic pollutants under visible-light illumination and ambient conditions to obtain a desirable mineralization process [44]. In the photocatalysis process, the reaction rate can be accelerated by using semiconductor photocatalysts to absorb light of the appropriate frequency [45]. Based on the phases of catalysts and reactants, the photocatalysis reaction may be heterogeneous or homogenous. It is identified as a homogeneous system if the catalyst and the reactant are in the same phase, while it is identified as a heterogeneous reaction if the catalyst and the reactant are in a different phase [46].

The photodegradation mechanism involves four main steps, including the absorption of light energy, excitation of electrons, separation of photo-carriers, and photocatalytic reaction [47-49]. By irradiating the photoreactor system with a light source (Eq. 2.1), the photocatalyst absorbs the photon energy equivalent to the frequency of its band gap energy (Eg), exciting the electrons (e−) from the valence band (VB) to the conduction band (CB), producing pairs of electrons and holes in the CB and VB, respectively [50,51]. The excited photo-carriers participate in a series of redox reactions to create a sufficient number of active radials.

Specifically, water molecules and/or hydroxyl ions can be reacted with splitter holes in the VB to generate hydroxyl radicals (•OH), as depicted in Eqs. 2.2 and 2.3. In addition, the superoxide radicals (•O_2–) and hydrogen peroxide (H_2O_2) can be obtained via a series of oxidation reactions between the photoexcited electrons of CB and dissolved oxygen (Eqs. 2.4 and 2.5), which convert to •OH radicals as illustrated in Eqs. 2.6 and 2.7 [52,53]. Subsequently (Eq. 2.8), the organic pollutants like dyes and antibiotics will be broken down by these ROS, converting them into simple and less harmful intermediates [54,55]. Unfortunately, the photoreaction can be hampered by reintegrating between the electrons and holes (Eq. 2.9), resulting in an undesirable treatment process [56,57]. Thus, it is important to overcome this deactivation action by using efficient modification technologies that will be clarified in the next sections" [58-60] (Figure 2).

$$\text{Photocatalyst} + \text{Light Energy}\left(h\nu\right) \rightarrow e^- + h^+ \quad (1)$$

$$h^+\left(VB\right) + H_2O \rightarrow \dot{O}H + H^+ \quad (2)$$

$$h^+\left(VB\right) + OH \rightarrow \dot{O}H \quad (3)$$

$$e^-\left(\text{CB of PC}\right) + O_2 \rightarrow \dot{O}_2^- \quad (4)$$

$$O_2 + 2H^+ + 2e^-\left(CB\right) \rightarrow H_2O_2 \quad (5)$$

$$\dot{O}_2^- + 2H_2O_2H\dot{O} + 2H\dot{O} + O_2 + H_2O_2 + \dot{O}_2^- \rightarrow \dot{O}H \quad (6)$$

$$H_2O_2 \text{ or } \dot{O}_2^- + e^- \rightarrow \dot{O}H \quad (7)$$

$$\text{ROS}\left(\dot{O}H, h^+, H_2O_2\right) + \text{organic} \rightarrow CO_2 + H_2O + \text{intermediates} \quad (8)$$

$$e^- + h^+ \rightarrow h\nu + \text{deactivated reaction} \quad (9)$$

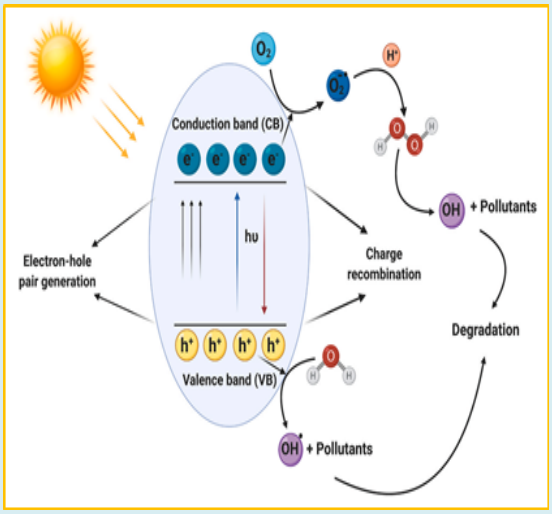

Figure 2: Schematic illustration depicting the photocatalytic mechanism under simulated sunlight irradiation [61].

Green Synthesis of Single Nano-Semi conductors

The green pathway refers to these methods that are employed to fabricate NP, which is characterized by being risk-free, less hazardous, environmentally friendly, and mostly employing renewable energy sources. In these technologies, plants and microorganisms are utilized as active bio-components in green synthesis, resulting in low energy consumption and environmentally friendly solvents, with water serving as the primary solvent. Besides, the NPs could be created by utilizing bioactive substances that serve as reducing and capping agents. The bioactive components are primarily sourced from various plant parts, including leaves, flowers, seeds, roots, and vegetable waste. Accordingly, bioactive phytochemicals are essential for the reduction of metal ions in the green synthesis of NPs. Generally, a simple method is employed for the manufacture of different kinds of metal nanoparticles mediated by plant extracts [62,63].

In the green synthesis approach, a known quantity of plant parts was washed three or four times with tap water and air-dried at room temperature. Then, the cleaned plant parts are cut into pieces (manually or with an electric grinder) and weighed before being added to 100 mL of distilled water at a high temperature for 20 min. After that, the extract is allowed to cool down to room temperature. To obtain a clear solution, the extract was filtered by Whatman filter paper No. 1 and stored at 0 to 4 °C. The filtrated extract was used as a reducing agent during the nanoparticle synthesis process

[64]. The deionized water is used to create an aqueous solution of metal precursors in varied concentrations. After that, the produced aqueous plant extract mixes in a different ratio with the obtained solution. The mixture was then stirred and heated to 80 °C. Bio-reduction can be detected by changing the solution color to dark brown. The produced nanoparticles can be centrifuged before being cleaned with deionized water and dried at room temperature. The final precipitate was annealed at high temperatures in a hot air oven for several hours before being cooled and collected for the next characterization step [64-66]. Sometimes, NaOH solution may be added to the mixture (metal precursors and plant extract) as an accelerator to increase the reducing potential and the rate of the reaction [67].

For instance [68], jujube fruit extract was used to prepare high-quality zinc oxide nanoparticles (NPs) using a green method. Moreover, ZnO NPs were fabricated using zinc nitride ($Zn(NO_3)2.6H_2O$) as a zinc precursor, and the jujube fruit aqueous extract was employed as an effective reducing agent and stabilizer. The ZnO NPs were applied for the degradation of two organic dyes (methylene blue (MB) and eriochrome black-T (ECBT)). The ZnO photocatalyst showed high photocatalytic activity, which was about 92% and 86% after 5 h of light radiation for MB and ECBT dyes, respectively. Moreover, the NPs demonstrated stable photocatalytic activity after sequential degradation experiments.

In another work, SnO_2 nanoparticles (NPs) were prepared by using Camellia sinensis leaf extract as a green method. In the synthesis procedure, three different extract concentrations (1, 2, and 4%) were utilized. The characterization technologies revealed that 4% SnO_2 NPs were different in average sizes and band gaps compared with the bulk sample. This result is attributed to the action of extracting molecules as a stabilizing agent. Moreover, the 4%SnO_2 NPs achieved a high degradation efficiency of MO (81%), and a total degradation of 100% of organic dyes, such as MB and Rd-B, after 180 min of UV-light radiation [69].

Moreover [70], silver nanoparticles (Ag NPs) were prepared using jujube core extract as a green, eco-friendly, facile, cost-effective, and rapid method. The photodegradation ability of Ag NPs was examined against cationic rhodamine B (RhB) and anionic eriochrome black T (EBT) pollutants under UV and visible light irradiations. The structural characterization of jujube core extract concluded that the biological activity of the extract can be attributed to its content of phenolic acids. The phenolic acids can stabilize and reduce the silver ions to silver nanoparticles. The photocatalytic experiments manifested that the degradation efficiencies were about 90.9% for RhB and 84.7% for EBT under UV irradiation and 74.7% for RhB and 68.2% for EBT under visible light irradiation within 80 min. The high degradation activity of Ag NPs against cationic dye compared with anionic dye could be attributed to the positive charge and oxygen group in the molecular structure of RhB.

In another work [71], green, facial, environmentally friendly, and simple biological procedure was used for the fabrication of selenium nanorods (Se-NRs) by adding ascorbic acid and gum Arabic (GA) to the aqueous solution of selenium precursor sodium selenite salt (Na_2SeO_3) (see Fig. 3). Briefly, ascorbic acid was used to reduce Na_2SeO_3 and synthesize Se nanorods, while gum Arabic was added as a stabilizing agent to prevent the undesired growth of Se-NRs and stimulate the force driver to control the particle size and improve the stability within the solution. The obtained Se-NRs displayed high stability and negative charge, as well as the shape of nanorods and a crystalline nature with an average size of about 24 nm. The photocatalytic performance of the obtained Se-NRs was evaluated in the elimination of RhB dye under UV light, which exhibited a degradation efficiency of about 85% within 120 min.

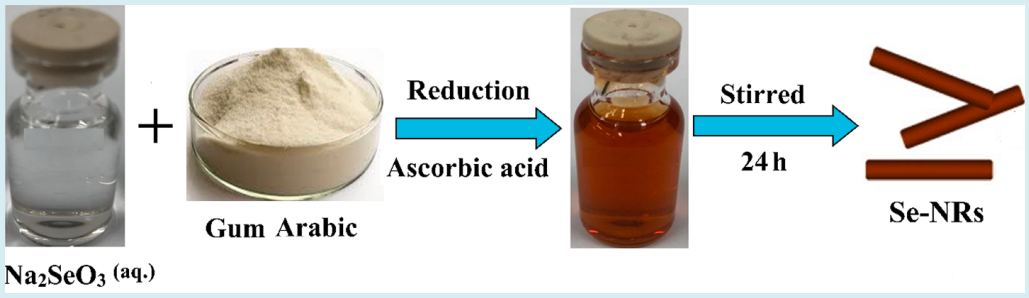

Figure 3: Green synthesis of Selenium Nanorods (Se-NRs).

In an interesting study [72], Cu-doped ZnO NPs were synthesized via bioreductant, eco-friendly, and Synedrium grantiid leaf extracts. Various concentrations of Cu were doped on ZnO NPs (1 to 9 wt%). The photocatalytic activity of the fabricated nanomaterial was studied against some organic pollutants like MB, Indigo Carmine (IC), and RhB under UV light. The X-ray photoelectron spectroscopy (XPS) analysis confirmed the existence of binding energies

between the host material and dopant ions. Moreover, when doping content increases, the band gap values also increase, negatively affecting the photocatalytic activity. Moreover, 3% and 5% Cu-doped samples exhibited enhanced degradation efficiency against the organic dyes. More impactful studies can be summarized in Table 1 (Figure 3).

No.	plant Name	Part of plant	NPs type	NPs Morphology	NPs Size (nm)	Organic pollutants	References
1	fenugreek (*Trigonella foenum-graecum*)	aqueous extract	Au NPs	spherical	20	4-nitrophenol	[73]
2	*Terminalia chebula* (*T. chebula*)	fruit	Ag NPs	face centered cubic geometry oriented in (1 1 1) plane	Diameter 25	methylene blue	[74]
3	*Plectranthus amboinicus*	leaf	ZnO NPs	Rod shape	88	methyl red (MR)	[75]
4	*Punica granatum*	fruit juice	gold and silver NPs	spherical	GNPs is 18 SNPs is 36	Methylene Blue (MB), Methyl Orange (MO) and Eosin Y (EY)	[76]
5	*Sterculia acuminate* (*S. acuminata*)	fruit	AuNPs	spherical	9.37 to 38.12	4-nitrophenol (4-NP), methylene blue (MB), methyl orange (MO) and direct blue 24 (DB24)	[77]
6	*Moringa oleifera*	flower	PdNPs	----	100	P-nitrophenol (PNP) and methylene blue dye.	[78]
7	*Carissa edulis* (*C. edulis*)		ZnO NPs	flower shaped	50–55	Congo red	[79]
8	*Zanthoxylum armatum*	leaves	Ag NPs	crystalline	range from 15 to 50	Safranine O, Methyl red, Methyl orange and Methylene blue	[80]
9	*Cicer arietinum*	leaves	Ag NPs	spherical	88.8	Congo red, 4-nitrophenol, and methylene blue.	[66]
10	*Hyphaene thebaica*	fruit	Ag NPs	spherical	20	4-nitrophenol (4-NP) and Congo red dyes (CR),	[81]
11	*Guiera senegalensis*	leaves	Ag NPs	spherical	50	Congo red dye (CR) and 4-nitrophenol (4-NP).	[82]
12	*Catunaregum spinosa* (C. *spinosa*)	root bark	Ag NPs	spherically	33±2	Amaranth dye	[83]
13	*Anacardium occidentale*	leaf	α-Fe_2O_3	nanocrystals	Less than 50	methyl red and eosin yellowish	[84]
14	*Camellia japonica*	leaf	Ag-NPs	spherical	around 12 to 25	nitrobenzene and EY dye	[85]
15	*Lagerstroemia speciosa*	leaf	Au NPs	spherical	41–91	methylene blue, methyl orange, bromophenol blue, bromocresol green, and 4-nitrophenol	[86]

16	*Cuminum cyminum*	seeds	Ag NPs	spherical	16 ± 2	Methylene blue (MB), methyl red (MR), rhodamine-B (Rh-B) and 4enitrophenol (4eNP).	[87]
17	*Tamarindus indica*	leaves	TiO_2	spherical	20-40	Titan yellow dye	[88]
18	*Trigonella foenumgraecum*	seed	Fe NPs	---	11	methyl orange dye degradation	[89]
19	*Mangifera indica*	flower	Ag NPs		Less than 51	4-nitrophenol and azo bond in dye molecules	[90]
20	*Allium ampeloprasum*	leaf	Ag NPs	quasi-spherical, spherical, ellipsoidal, hexagonal and irregular	between 2 and 43	4-nitrophenol to 4 aminophenol, DPPH• (2,2-diphenyl-1-picrylhydrzyl) and ABTS+• (2,2'-azino-bis (3-ethylbenzothiazoline-6-sulphonic acid)) radicals.	[91]
21	*Alcea rosea*	leaf	Au NPs	triangular, pentagonal, hexagonal and spherical	Apr-95	4-nitrophenol pollutant	[92]
22	Sal deoiled seed cake (DOC), a plant-based waste as reducing capping agent	Seed	Ag NPs	polygonal	30–150	namely Methyl orange, Congo red, Methylene blue, Eriochrome black T, and Evans blue.	[93]
23	Hibiscus *Rosa sinensis*	leaf	$ZnCr_2O_4/ZnCrO_4$	crystalline	70-14		[94]
24	corn-cob	waste	CC-Ag NPs CC-AuNPs	Spherical multiple shapes	2 to 28 5 to 50	o-, m-, p-nitrophenols, , Rhodamine 6G, and Eosin Y.	[95]
25	*Terminalia bellerica*	kernel	Ag NPs	----	Less than 48	4-nitrophenol, methylene blue, eosin yellow and methyl orange.	[67]
26	*Clitoria ternatea*	flower	SnO_2	spherical	7	Rhodamine B (RhB)	[96]
27	*Ceratonia siliqua*	----	Fe_3O_4-cellulose-copper nanocomposite	spherical	25	4- nitrophenol, 2,4-dinitrophenylhydrazine, methyl orange and potassium ferricyanide.	[97]
28	jumbo Muscadine (*Vitis rotundifolia*)	the epicarp (cover) and endocarp (seeds)	GCoO-NPs	-------	--------	AB-74 dye	[98]
29	*Cydonia oblonga*		NiO-NPs	cubic shape	dimension (74.5 nm)	Rhodamine B (RB) dye	[29]
30	*Camellia sinensis*		SnO_2	quasi-spherical	6.91, 5.2, and 4.7	dyes Methylene Blue (MB), Methyl Orange (MO), and Rhodamine B (Rd-B)	[69]
31	*Citrus reticulata blanco*	peel	Ag NPs	spherical	Less than 27	malachite green dye	[65]
32	*Melia azedarach*	Leaf	Cu-ZnO	irregularly shaped particles		chlorpyriphos pesticide	[99]

33	*Canna indica L.*	Flowers	ZnO	crystallite	27.82	methylene blue	[100]
34	*Solanum torvum* (Turkey Berry)	Fruit	Au NPs	spherical	9–14	Methylene Blue dye	[101]
35	*Vernonia amygdalina*	Leaf	α-Fe_2O_3	resemble ginger	----	methyl orange (MO) and methylene blue (MB) dyes	[102]
36	*Dodonaea viscosa*	plant species	Ag NPs	---	60	4-nitrophenol (4-NP), 2-nitrophenol (2-NP), and azo dyes included Rhodamine B (RB), Congo red (CR), and Methyl orange (MO).	[103]
37	*Eucalyptus globulus*	outer fruit shell	Ag NP	----	-----	Methyl orange, methyl red and congo red	[104]

Table 1: Examples of NP synthesis with plant assisted and their performance in photocatalytic degradation of organic pollutants.

Green Synthesis Heterojunction Photo catalyst

Photocatalytic processes by nanomaterials can be considered one of the most creative techniques because of their ability and flexibility for manipulation to enhance their activity. Coupling two nano-semiconductors in one system has been demonstrated to be one of the most efficient ways to boost the photocatalytic capacity [105]. The enhancement by heterojunction construction is coming by overcoming the drawbacks of single photocatalysts like fast recombination of photo-generated electron-hole pairs, limited electron mobility, restricted optical absorption, or insufficient active sites [106]. According to the electron transfer pathway between the contributed nanomaterials in one heterojunction, the green synthesis heterojunction has many types, like nanoparticle/semiconductor heterostructure, plasmonic heterojunction, etc.

For example, a ternary Ag/RGO/Fe_3O_4 heterogenous catalyst was biosynthesized using Lotus garcinii leaf extract. The plant extract was used as an agent for the reduction and stabilization of silver ions from the precursor's solution ($AgNO_3$) to silver NPs on the RGO/Fe_3O_4 nanocomposite. The photocatalytic performance of the prepared nanocomposite was assessed using many organic pollutants, including 4-nitrophenol (4-NP), CR, and RhB. The green synthesis of NPs can greatly reduce their adverse environmental effects. Moreover, RGO acted as a matrix in the heterojunction, preventing the accumulation of NPs, and was magnetized to facilitate the separation of nanocomposite from the reaction solution. The Ag/RGO/Fe_3O_4 heterojunction present excellent photocatalytic efficiency in 180 min [107].

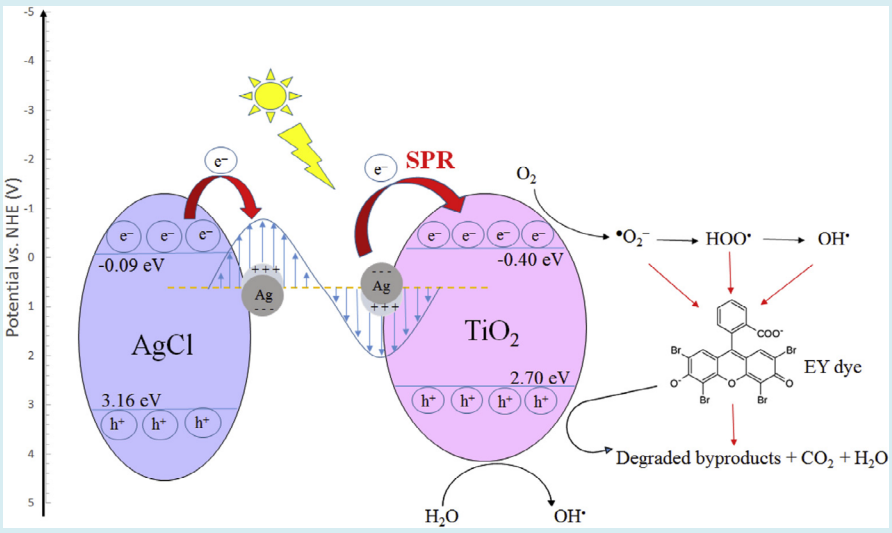

Figure 4: The band structures and photodegradation mechanisms of the Ag/AgCl@TiO2 heterostructure under sunlight.

In another study [108], mangrove plant Avicennia marina aqueous leaf extract was used in the facile biosynthesis of Ag/AgCl@TiO_2 plasmonic heterojunction. The extract was applied as a reducing and stabilizing agent as well as a chlorine source to synthesize Ag/AgCl from $AgNO_3$ precursors. The characterization results revealed successfully synthesized Ag/AgCl@TiO_2 nanocomposite with nanosized particles (~35 nm), high specific surface area (31.94 m^2/g), and improved bandgap energy (1.73 eV). The photocatalytic activity of the prepared Ag/AgCl@TiO_2 was examined against the degradation of eosin Y dye under sunlight radiation. The photocatalytic activity indicated high removal efficiency (99.74% for 50 ppm dye concentration, 50 mg/100 mL catalyst dose, and 4 pH at 35°C) after one hour of sunlight radiation. This high performance was attributed to the surface plasmon resonance and the effective charge separation due to the heterostructure developed among Ag, AgCl, and TiO_2, as shown in Figure 4.

Conclusion

A few days ago, organic pollutants revealed a greater threat to human health and the environment due to the potential contamination of marine life and ecosystems. A huge number of metal oxides, such as TiO_2, WO_3, SnO_2, CeO_2, ZnO, Bi_2O_3, Ag-based photocatalysts, Nb_2O_5, and so on, were employed as semiconductor photocatalysts for the degradation of organic pollutants under UV and visible light sources. This review introduced valuable information about the fabrication of nanoparticles via green synthesis approaches. These methods demonstrated promise as technologies due to being risk-free, less hazardous, environmentally friendly, and mostly employing renewable energy sources. The synthesized nanoparticles revealed robust photocatalytic activity for the degradation of different organic pollutants under UV and visible light sources. This study discussed recent advances and the sustainable fabrication approach to developing stable nanoparticles. Moreover, the preparation of green heterojunctions and their application in wastewater treatment were also highlighted.

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