Xi'an Shiyou Daxue Xuebao (Ziran Kexue Ban)/
Journal of Xi'an Shiyou University, Natural Sciences Edition
ISSN: 1673-064X
E-Publication: Online Open Access
Vol: 67 Issue 03 | 2024
DOI: 10.5281/zenodo.10867778

TRENDS AND LATEST ADVANCEMENTS IN THE SYNTHESIS METHODS IN PHOTOCATALYSTS

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Abstract

Access to clean water is vital for human survival and societal advancement, especially amidst global challenges like environmental degradation. Photo catalytic technologies are increasingly prominent in wastewater treatment. The synthesis process significantly influences the properties and effectiveness of photocatalysts. This article focuses on various synthesis techniques, including synthetic and green methods, aiming to produce efficient and affordable catalysts for purifying water contaminated with dyes, pesticides, and other pollutants. Examples of recent photocatalysts highlight diverse approaches. Synthesis methods range from sol-gel to electro-deposition, with green synthesis gaining traction due to its environmental benefits. Biogenic approaches utilizing plant extracts and microorganisms offer sustainability and scalability. The article emphasizes recent advances in green synthesis, particularly in producing undoped and doped nanomaterials for water treatment. It also discusses future trends in synthesis methods and research directions, underscoring the importance of sustainable approaches in addressing water pollution challenges.

Keywords: Photocatalysts, Recycling Wastewater, Biogenic Approaches, Green Methods, Organic Dyes, Synthetic Methods, Catalysts.

1. INTRODUCTION

Rising urbanization and rapid industrialization in developing countries results in the skyrocketing demand of clean water in huge quantity that redirected to the households, commercial, and industrial sectors, resulting in the generation of more wastewater. Pollutants such as dyes, toxic solvents, chemicals, metals, oils, and so on are discharged by companies, turning the water unfit for human consumption. Unwanted pollutants in wastewater can be detrimental to the health of people as well as the environment[1-3]. According to World Bank report 2020, despite the environmental, health, economic, and financial benefits of reusing wastewater, the 80% of it is released into the environment without appropriate treatment globally. According to REN21's Renewables in Cities Global Status Report, from 2009 and 2019[4-5], renewable energy only accounted for 25% of the rise in overall energy consumption (see Fig. 1) (see Fig. 1).

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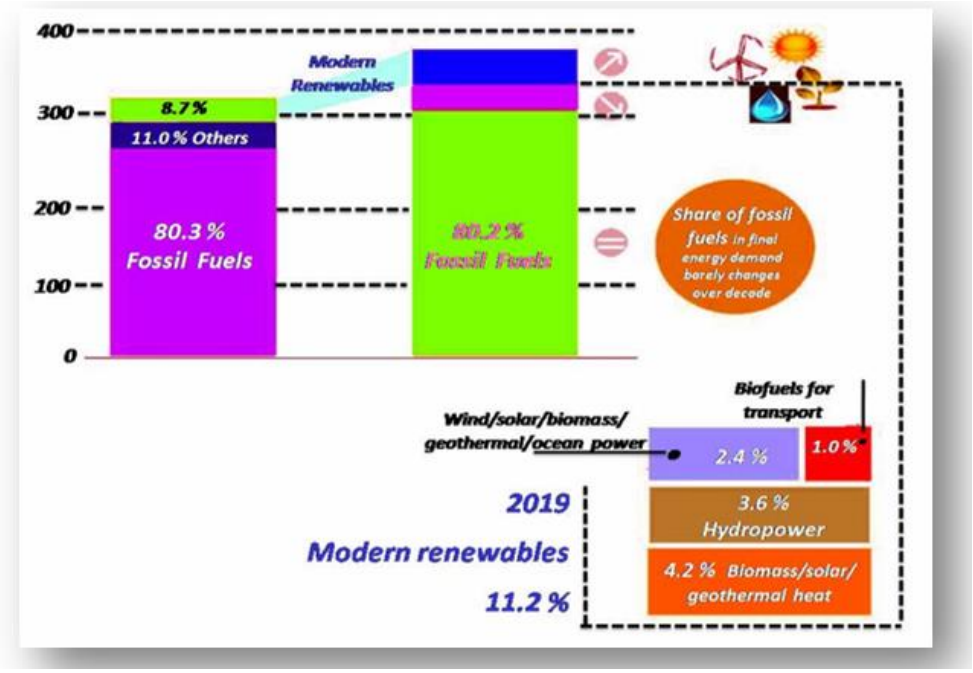

Fig 1: Anticipated Ratio of Renewable Energy to the Total Final Energy Consumption over a Ten-Year Period, 2009–2019

The practise of not reusing and recycling wastewater is becoming increasingly unsustainable and unscrupulous[6]. The most effective method to address the issue and to alter our viewpoint on how we consume and recycle waste. The obvious solution to address this vicious spiral of the ecological issues is to lay its foundation on some renewable energy technology. Photocatalysis being a prominent renewable energy technique that utilizes light to generate energy through artificial photosynthetic reactions namely water splitting and CO_2 reduction[7-8]. Eibnor, a German scientist, first, utilised this method developed in 1911[9] to use ZnO in the presence of sunlight to bleach dark blue pigment. Further, in 1938, after the discovery of TiO_2, photocatalysis was utilized as a water purification technique for organic dye pollutants under UV irradiation by employing the advanced oxidation processes (AOPs)[10]. Baly et al. introduced the term photocatalysis in the title of a publication in 1921, but the meaning attached to it was associated with instances where light acted as a catalyst for a reaction[11].

The concept of "photocatalysis" was introduced by Deerffler and Hauffe in 1964 to indicate that a reaction might be changed by the combination of light and a solid catalyst[12–13]. Subsequently, Fujishima and Honda introduced a study in 1972 on the photocatalytic water splitting on TiO2 electrodes under UV irradiation, signalling the inception of photocatalysis research[14].

Nanotechnology advancements further paved the path for the invention of novel types of photocatalysts for future sustainable technologies. Nanotechnology blesses us with enormous potential that can be utilized for upgrading wastewater and water treatment,

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both in terms of improving treatment and expanding the availability of water by safely using scarce water sources. It spans a number of disciplines because it is an interdisciplinary area, including environmental science, biology, physics, engineering, chemistry, and medicine[15-16].

Due to their high surface area to volume ratio, photocatalysts made of the same material at the nanoscale exhibit characteristics that are significantly different from those of the bulk state. effects, tunable photo activity, increased strength, and wide range applications[17-18].

The factors to be kept in mind while synthesizing a photo catalyst are[19-20]:

- The effective light absorption capability of the photocatalyst for stimulating electrons from the valence band (VB) to the conduction band (CB).
- The photo-efficiency of the photocatalyst.
- The charge separation ability of photo-generated electron-hole pairs.

2. FABRICATION OF PRACTICAL PHOTOCATALYSTS

Photocatalysis is a significant step towards the development of green and eco-friendly, non-toxic, and economically viable technology. A key objective of nano catalysis research is to create catalysts boasting 100% selectivity, outstanding activity, low energy requirements, and prolonged durability.

Achieving this necessitates meticulous control over factors such as size, shape, spatial arrangement, surface composition, electrical properties, as well as thermal and chemical stability of nanostructures[21-22].

Faccani et al. synthesized TiO_2 coated fabric (photocatalyst) for the photo-degradation of Rhodamine B (RhB) dye. This research expanded the application of TiO2-based photocatalysts to utilize solar energy, making them suitable for areas lacking electricity or as a sustainable alternative to avoid the use of bio-hazardous and expensive UV light sources[23].

However, if these synthesised nanomaterials are subjected to practical applications, they may face the following constraints such as stability in challenging situations, waste recycling, toxicity characteristics, lack of understanding in profound mechanism and modelling factors, need for skilled professionals, and extensive analysis requirements. So, we need to adapt the synthesis methods for the photocatalysts, keeping in mind the above-mentioned problems.

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3. TYPES OF SYNTHESIS METHODS

Currently a considerable number of nano catalysts are available because of the numerous types of synthesis techniques. From literature survey, it has been found that photocatalysts with different sizes, morphologies, and compositions can result in manifold photocatalytic characteristics[24].

An extensive range of synthesis methods can achieve this versatility. The synthesis of nanomaterials is done via following two approaches: bottom-up or top-down. Macroscopic particles on a large scale are transformed into nano-scale particles using a top-down approach for producing structures with long-range order. It includes sputtering, thermal/laser ablation, mechanical/ball milling, etching, etc.[25-27].

However, this procedure is cumbersome, time-consuming, and costly. In the bottom-up approach, nanoparticles are produced through physico-chemical and biological methods to assemble at the nanoscale, creating short-term order. It includes co-precipitation, sol-gel, hydrothermal/solvothermal, chemical vapor deposition (CVD), laser/ spray pyrolysis methods, among others[28-31].

For decades, physical and chemical approaches have been used to create nanomaterials. Traditional physicochemical methods for synthesizing nanomaterials often involve the use of hazardous and volatile substances.

Consequently, there is a growing impetus among researchers to devise eco-friendly, safer, and more cost-effective green approaches for creating innovative and viable nano catalysts capable of removing and degrading a wide range of water pollutants.

For the production of nanoparticles with well-defined shapes, a physical process involving physical force, expensive apparatus, and extreme temperature and pressure is used. Chemical procedures, on the other hand, use harmful ingredients and result in noxious by-product evolution through the synthesis course. Bottom-up approach has higher precision, accuracy and control over the surfaces and edges of the synthesized nanostructures than top-down approach.

Green synthesis process is one of the bottom-up approaches in the nano-technological era since it is low cast and affordable and environmentally friendly, with minimum by-product evolution.

It is an alternate way that can be opted by making use of microorganisms (fungi, bacteria, and algae), plant tissues (leaf, stem, root, fruit, peel, and flower) and biomass as shown in Fig. 2.

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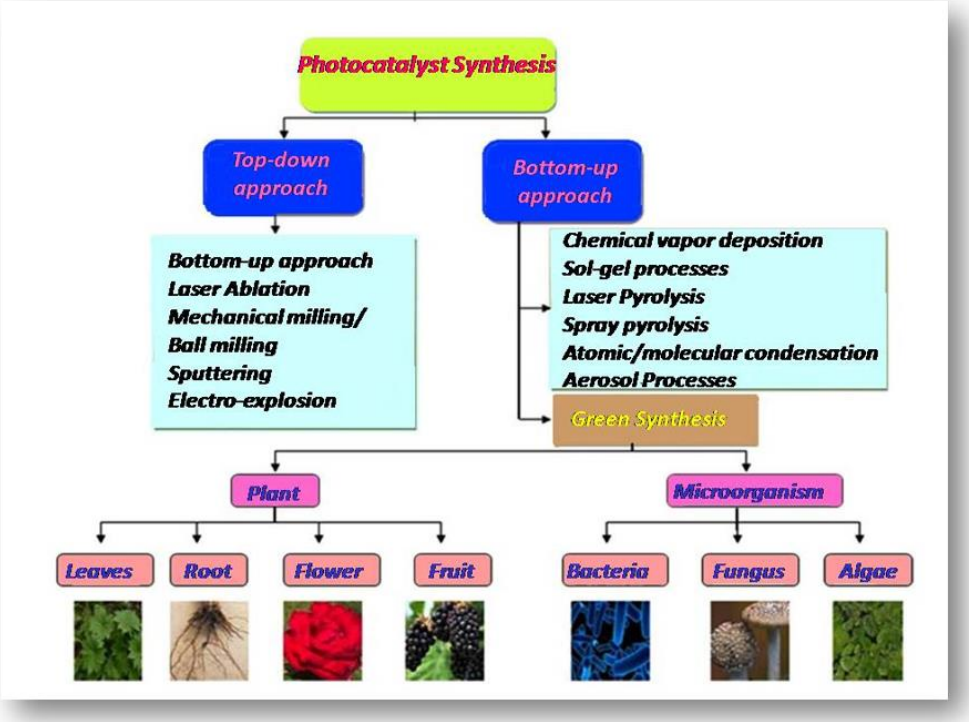

Fig 2: Classification of various synthesis methods for photocatalysts

The present article explores emerging trends and forthcoming prospects in employing green-synthesized nano catalysts and nanomaterials for water and wastewater treatment. This encompasses the development of advanced nanomaterials and novel nanoscale adsorbents using eco-friendlier and sustainable methods to remove pollutants and ions from various water-based solutions, including groundwater, drinking water, and wastewater. It also delves into the current advancements, future challenges, and potential applications of eco-friendly nano catalysts and nanomaterials in the realm of wastewater treatment and purification.

Embracing the advancement of environmentally conscious treatment technologies is vital for industries dealing with hazardous and chemical-laden wastewater.

4. GREEN PHOTOCATALYST SYNTHESIS: SUNLIGHT-ACTIVE PHOTOCATALYSTS

Rising energy demand, environmental pollution, and more strict environmental policies have made the quest for alternative green synthetic methods and the development of sustainable technologies unavoidable at the moment. Given that the sun consistently cast light on the earth with 120,000 terawatts of energy, which is over 6000 times the Earth's energy consumption, hence, it can be used for green synthesis technology. Photochemistry is an extremely promising technique since it employs solar light (which contains approximately 5% UV, 43% visible, and 52% infrared) as a sustainable,

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inexhaustible, and sustainable energy source[32-33]. The concept of "green" synthesis has garnered significant attention as a dependable, sustainable, and eco-friendly approach in materials science for producing a diverse array of photocatalytic nanomaterials. These include both undoped and doped metal/metal oxide nano catalysts, hybrid nano catalysts, sunlight-active nano catalysts, and bioinspired nano catalysts. The green photocatalysis is a synthesis route that aids in the manufacture of various photocatalysts by the use of natural resources, biomasses, and biological extract[34-35]. Hence, it is recognised as a significant tool for mitigating the negative effects connected with the standard methods for the synthesis of nanostructures used in laboratories and industry.

The absorption of a broad limit of the solar spectrum, ability to dissociate water molecules, and stability in water environment during the reaction processes are some of the prerequisites for an ideal photocatalyst. Moreover, it needs to be financially viable, easy to handle, readily accessible, and environmentally safe. In recent decades, numerous metal oxide semiconductor-based nanostructures have been developed and validated as photocatalytic agents for water treatment, harnessing specific segments of the solar spectrum[36-37]. Among extensively studied semiconductors for potent photocatalysis, tailored transition or d-block metal ions have demonstrated remarkable efficacy.

The factors on which the photocatalytic efficiency depends include:

- Concentration of dye and photocatalyst
- pH of dye solution
- Reaction temperature
- Intensity of light and period of irradiation
- Surface morphology and surface area of the photocatalyst
- Doping of photocatalyst

4.1. Metal and Metal oxide Photocatalysts

Most of the semiconductor photocatalysts in use could only absorb photons with a bandgap greater than the bandgap of the photocatalyst. However, the bandgaps of commonly used TiO_2 and ZnO are roughly 3.6 and 3.2 eV, respectively, resulting in absorption in the UV region of electromagnetic spectrum for these materials (UV ~ 5% of the whole solar spectrum)[38-39].

As a result, researchers have been finding strategies to improve absorption ability, both in terms of widening the wavelength range and increasing absorption intensity of the semiconductor photocatalysts. Bandgap engineering, doping, shape and size modification, and loading with metal or non-metal particles are only a few of the ways that have been discovered to create extremely efficient photocatalysts[40-41]. The coupling of semiconductors with metal nanoparticles, particularly noble metal nanoparticles, has recently drawn a lot of interest among the various approaches[42-43].

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Three metal forms may usually be considered based on the diverse enhancing processes of metals on semiconductor photocatalysts: doping ions, nanoclusters, and plasmonic nanoparticles. Due to single-step and economically beneficial synthesis process, the creation of silver nanoparticles (Ag NPs) using a green approach has gotten a lot of interest in recent times. Vijay et al. found that Ag NPs obtained from Boerhavia diffusa plant extract had a spherical-shaped morphology with a diameter of 25 nm and have better bacterial properties against fish pathogens[44].

Nagajyothi et al. found gold nanoparticles of various shapes using Lonicera Japonica flower extract, including face-centered cubic, quasi-spherical, triangular, and hexagonal, with an estimated mean size for the AuNPs of 8.02 nm[45]. Huo and coworkers used the stem extract of Chenopodiumaristatum L. to create AuNPs with spherical, truncated triangular, hexagonal, pentagonal, rod-like, and irregular forms with a yield conversion of more than 99 %[46].

Based on the phytochemicals included in the produced extracts, the above-mentioned preparation methodologies suggest that each plant extract with varied amounts contributes to the various size and morphological properties of Au NPs as shown in Table 1.

Apart from gold, silver, or platinum nanoparticles, Cu nanoparticles have received a lot of attention as it is economic. Sinha et al. found that spherical Cu nanoparticles were synthesized by a single- step green process using fish scales of Labeorohita with an average size range of 25-37 nm[47].

Upon mixing fish scale extract with copper sulphate solution, an instant complex formed due to the electrostatic attraction between the negatively charged gelatin and the positively charged Cu^{2+} ions. It is because of the collagen-rich L. rohita and some of the primary components in the fish scale extract, such as glycine, amino acids, hydroxyproline, and hydroxyzine[48].

Among several semiconductor metal oxides TiO_2, ZnO, SnO_2, Cu_2O, and WO_3 with these parameters have alike photocatalytic performance, for instance light absorption. This stimulates photogenerated charge carriers, resulting in the formation of voids, capable of oxidising organic compounds. The essential characteristics for an efficient photocatalytic material include the necessary band gap, optimal band edge position, large surface area, precise morphology, chemical stability, and the ability to be reused.

Metal oxide NPs are synthesized in a variety of ways, including physical, chemical, and biological methods, but green production of metal oxide NPs. Plants, bacteria, fungus, seaweed, and microalgae have all been found to be successful in the synthesis of various metal and metal oxide nanoparticles[49-52].

Plant extracts have gotten a lot of interest recently due of their ease of use, low cost, and quick reaction time, as well as their potential to reduce metal ions to metal nanoparticles and generate nanoparticles on a big scale.

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Nethravathi and his coworkers reported facile green synthesis of multifunctional ZnO NPs using water extract of Garcinia xanthochymus (fruit) via solution combustion synthesis and discovered that the plant components act as antioxidants which can further be used as reducing agents[53]. Vidhu et al. reported the biogenic synthesis of SnO2 NPs using Saracaindica flower extract as reducing agent[54].

In 2020, Rafique et al. found that CuO NPs synthesized using novel leave extract of Citrus aurantifolia exhibited excellent photocatalytic degradation of RhB dye (91%) and antibacterial properties towards gram positive S. aureus and gram-negative E. Coli bacteria[55]. In 2021, Kahsay synthesized ZnO NPs using aqueous leaf extract of Beciumgrandiforum in which the phytochemicals such as s phenols, flavonoids, saponins, glycosides, steroids, tannins, and alkaloids were used as capping and reducing agent[56].

Table 1: Overview of metal/ metal oxide green-synthesized nanoparticles from various plant extracts and their morphologies

Photocatalysts	Biogenic Source	Morphology	References
Au NPs	Citrus lemon (fruit extract)	Spherical	57
Au NPs	Momordicacochinchinensis (Lour.) Spreng (leaf extract)	Spherical, oval, and triangular	58
Ag NPs	Banana peel extract	Spherical	59
Cu NPs	Plantagoasiatica (dried leaf powder)	Spherical	60
CO NPs	Syzygiumalternifolium (bark extract)	Spherical	61
ZnO NPs	Eucalyptus globulus (dried leaf extract)	Spherical	62
ZnO NPs	Araliaceae family (root extract)	four-way leafy flower	63
CeO_2 NPs	Aloe vera (leaf extract)	Spherical	64
Fe_2O_3 nanorods	Use of a natural resin (Musa Paradisiaca Linn) as an oxygen source and stabilizing agent	Spindle-shaped nanorods	65
SnO2	Catunaregamspinosa (C. spinosa) root barks	Spherical	66

4.2 Doped Metal and Metal oxide Photocatalysts

Doping represents an optimal method for enhancing the absorption capacity and manipulating the electrical properties of a semiconductor photocatalyst. Doping can also improve the charge transmission properties of semiconductors, resulting in increased carrier transfer efficiency.

The dopant's redox potential, surface characteristics and ionic radii have a significant impact on the photocatalytic efficiency. Metal and non-metal doping have garnered considerable interest in recent years. (See Fig. 3).

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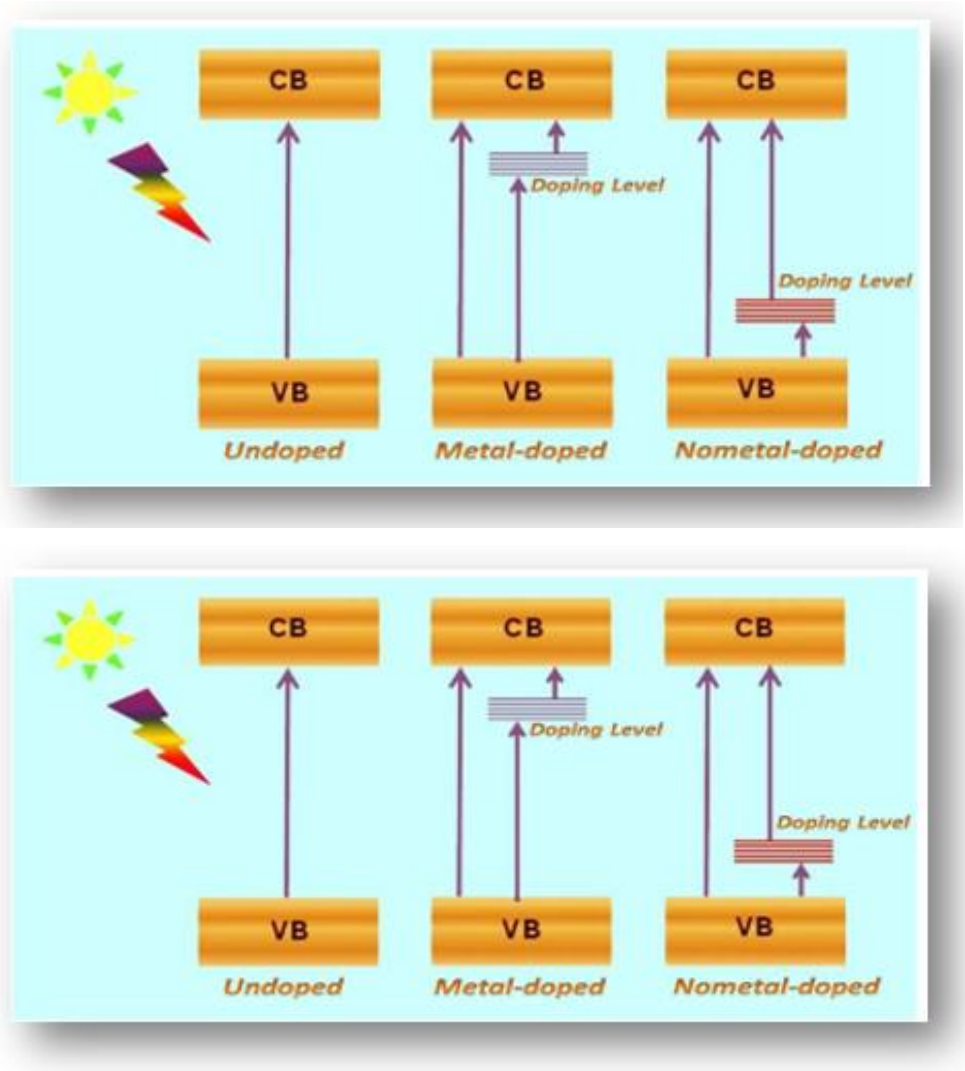

Fig 3: Energy levels for undoped and doped (metal/non-metal) photocatalysts

Extensive research has been conducted on doping with non-metal ions (such as carbon, nitrogen, boron, and sulfur) to amend the bandgap and band edge station of a semiconductor, enabling it to function as a visible-light active photocatalyst[67-68]. Doping metallic metals such as Au, Ag, Co, and Cu into semiconductor oxides can boost their photocatalytic activity and induce a narrowing bandgap, causing the photo-absorption edge to migrate toward the visible area.

Variations in photocatalysts are used to prolong electron-hole recombination, broaden the absorption spectrum, increase photostability, and speed up reactions on the catalyst's surface (see Table 2). Atarod et al. reported the facile green synthesis of Ag/TiO_2 nanocomposite by extract of leaves of Euphorbia heterophylla in which flavonoids present act as both reducing and capping/stabilizing agents[69]. In 2016, Kulkarni et al. synthesized

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Ni-MgO synthesized by facile green synthesis using Synadeniumgrantii plant extract showed 94% photocatalytic efficiency for MB dye under UV light irradiation while undoped MgO showed 85%[70].

Alharthi et al. conducted a study on the eco-friendly synthesis of Ag-ZnO nanoparticles utilizing potato wasteland via a straightforward and cost-effective combustion method, varying dopant concentrations. Their findings revealed that nanocomposites containing 2 percent Ag-ZnO exhibited the highest catalytic activity (96%) in degrading MB dye within 80 minutes under visible light irradiation. This outcome was attributed to the limited availability of Ag for electron absorption by the conduction band (CB) of ZnO[71].

Table 2: Overview of green-synthesized nanoparticles doped with metal oxides

Photocatalysts	Biogenic Source	Application	References
Ag/CuO	Use of leaves extract (Cyperuspangorei) as a stabilizing and reducing agent	Dye degradation, antibacterial activity	72
Pd/CuO	Theobroma cacao L (seed extract)	Dye degradation	73
Zn-TiO2	Use of plant extract (Green Tea) as a reducing agent	Dye degradation	74
Au-ZnO	Use of leaves extract (Caryaillinoinensis) as a stabilizing agent	Dye degradation	75
Co/ZnO	Use of Eichhorniacrassipes plant tissue for the accumulation of Co and sequential combination with ZnO	Dye degradation	76

4.3 Hybrid Photocatalysts

Wide bandgap photocatalysts encounter the challenge of swift recombination of photo-induced charge carriers. However, a lower bandgap, which enhances light harvesting capabilities, is required to improve solar energy use efficiency at the same time[77]. As a result, the development of simple ways to overcome these obstacles is critical for boosting photocatalytic performance. Two semiconductors can thus be coupled in such a way that the (VB and CB) band location edges of two photocatalysts are staggered in relation to one another[78]. Till date, a lot of effort has gone into resolving these problems. The creation of heterojunction devices, among other methodologies, is promising due to its merits in separating photogenerated electron–hole pairs and linking the relative advantages of each component. Two semiconductors with staggered band structures are always used to form heterojunctions, and the Type-II photocatalytic mechanism is frequently used on these heterojunctions. (Fig. 4)

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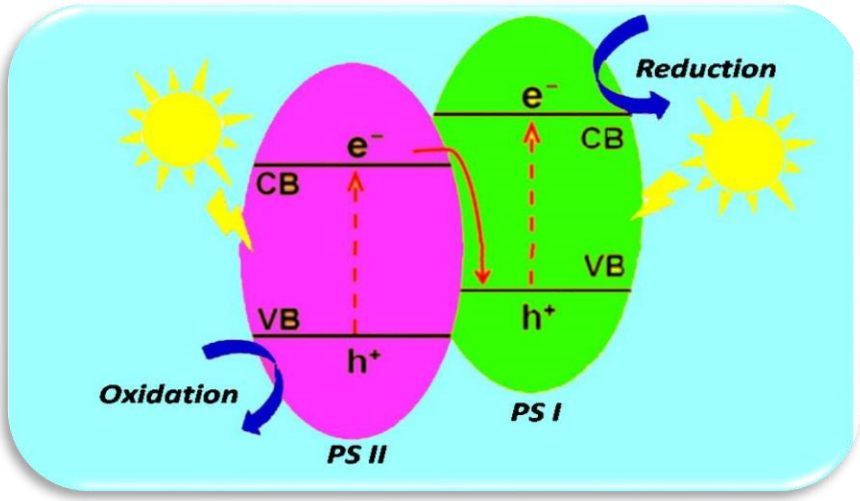

Fig 4: Direct Z-Scheme Mechanism for Type-II Heterojunction Photocatalysts

When light strikes PC I and PC II, it generates photogenerated charge carriers in both photocatalysts, as illustrated in Fig. 4. The photogenerated electrons from PC II, which possess a low reduction potential, tend to recombine with the photogenerated holes from PC I, having a low oxidation potential. This process leaves behind photogenerated charge carriers in both PC I and PC II at their respective redox potentials, facilitating spatial separation and enhancing the redox ability of the photocatalytic system.

In comparison, charge carrier migration is more favorable in the direct Z-scheme photocatalyst than in the type II heterojunction. The electrostatic attraction between electrons and holes promotes electron migration from the conduction band (CB) of PC II to the hole-rich valence band (VB) of PC I, which is thermodynamically advantageous. Consequently, the direct Z-scheme system exhibits superior photocatalytic performance[79-80]. Theelectron maintains a lower negative potential whereas the hole maintains a larger positive potential, resulting in the system's stronger redox capability. Not only does this system improve charge spatial separation, but it also maintains a larger redox potential[81].

Ramanathan et al. synthesized SnO_2-C. annuum reduced graphene oxide (CRGO) nanocomposite using Capsicum annuum (CA) as reducing agent. As CRGO and SnO_2 nanoparticles are combined, CRGO/SnO_2 nanocomposite has a better photodegradation efficiency than other materials. The nanocomposite's reduced surface area has increased dye adsorption, while CRGO traps excited electrons, reducing recombination and increasing photodegradation efficiency (97.4%/ 60 min)[82]. Djellabi et al. synthesized the magnetic visible light-responsive TiO2-OP@Fe3O4 nanocomposite demonstrates enhanced photocatalytic activity compared to TiO2-OP and TiO2@Fe2O3. Photocatalytic tests reveal that TiO2-OP@Fe3O4 exhibits efficient adsorption and performance in degrading dyes (MB, RhB, and CR) and reducing Cr (VI)[83].

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5. CONCLUSION

The realm of photocatalysis holds vast promise for nanotechnology, leveraging solar energy to mitigate organic pollutants in the environment. To address advanced environmental concerns, safe, green, and convenient methods of synthesizing nano catalysts are required. This article aims to discuss the recent trends and advancements in the synthesis methods in photocatalysts as well as new research directions. Article is centric over the possible synthesis techniques for developing photo catalytic materials and their potential in treating water samples contaminated with foreign elements. Herein, focuses were made on discussing green synthesis technique for reducing the harmful consequences associated with standard synthesis methods for photo catalysts and highlighting the topical expansion of green synthesis pathways for photo catalyst synthesis.

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