IJARSCT
ISSN (Online) 2581-9429
International Journal of Advanced Research in Science, Communication and Technology (IJARSCT)
International Open-Access, Double-Blind, Peer-Reviewed, Refereed, Multidisciplinary Online Journal
Impact Factor: 7.53
Volume 4, Issue 2, September 2024

A Comprehensive Review on Synthesis Techniques of Nanocomposites for Visible Light-Active Semiconductors

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Abstract: *The photocatalysis of different semiconductors has been the focus of several investigations on the breakdown of harmful organic compounds in waste water. Due of its potential to address several contemporary environmental issues, including pollution of the air and water, it has garnered significant interest in modern research. Because of their great photosensitivity, stability, and lack of toxicity, nano composite metal oxides, which make up the majority of typical light catalysts, are well-known for their efficacy in degrading a variety of environmental pollutants. Different methods exist for synthesising nanomaterials, including: both top-down and bottom-up methods. The optimal method is called the "top-down approach," which involves cutting a bulk material piece by piece until nanoparticles are obtained. Controlling the size, shape, distribution, composition, and degree of aggregation of the particles is of importance to these applications. The use of certain nanoparticles for photocatalytic applications is limited by their huge band-gaps, which cause a high rate of photogenerated electron-hole pair recombination. In recent times, noteworthy efforts have been undertaken to create novel or altered semiconductor photocatalysts that can use visible light (λ = 400 nm-700 nm). These endeavours include semiconductor coupling, metal ion doping, nonmetallic element doping, and organic dye sensitisation. Numerous studies have shown that coupling two semiconductor nanoparticles with differing band gap widths is one of the best strategies to lower electron-hole pair recombination and, as a result, increase photocatalytic activity. In addition, the ternary nano composites exhibit significant photocatalytic activity in visible light and effectively degrade organic dyes, suggesting a greater level of photocatalytic activity.*

Keywords: Coupling, Doping, Nanocomposite, Photocatalyst.

I. INTRODUCTION

The textile industry' dye effluents discharge hazardous and possibly cancer-causing compounds into the water, leading to serious environmental pollution issues. Water is essential to human existence since it is used in both industrial and agricultural processes. Both environmental chemistry and water technology are quite interested in wastewater treatment. Consequently, it is even more crucial to create affordable and efficient dye wastewater treatment techniques. The colour of the dye-contaminated water is now being degraded by primary secondary and chemical processes approaches using standard technologies. These methods, however, are non-destructive since they only shift the non-biodegradable material into sludge, creating a new kind of pollution that requires further treatment.

The Active Oxidation Process technology has garnered great interest from many segments of the scientific community due to its ease of handling and notably lower residuals when compared to conventional procedures. The UV photolytic procedure, photo-fenton process, zonation process, sonolysis, photocatalytic approach, and radiation-induced dye degradation are a few of the several approaches used in the AOP approach. Many recent research have focused on the photocatalysis of different semiconductors to degrade harmful organic chemicals found in waste water.

Strong, high surface area heterogeneous photo-catalysts and catalyst supports made of nanoparticles have replaced traditional bulk materials as sustainable options. The high surface to volume ratio of the nanoparticles increases the catalyst's active component's exposed surface area and improves the catalyst-reactant interaction. Surface area is thus

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crucial to photocatalytic activity; as a result, attention has been drawn to semiconducting nanomaterials owing to their high surface to volume ratio. For the redox processes to take place for the contaminant to fully degrade or mineralise, a contaminant molecule must be adsorbed on the surface of the photocatalyst. Although large band-gap semiconductor nanoparticles with special structure and characteristics have been used in photocatalysis, their use is limited by the rapid rate at which photogenerated electron-hole pairs recombine. As a result, efforts have been made to enhance the photocatalytic performance of nanoparticles using a variety of techniques, including the synthesis of one-dimensional nanorods, nanorod arrays, combination with other components, transition metal addition, non-metal doping of metal oxide nanoparticles, and use of binary and ternary semiconductors.

The two distinct energy level systems of the connected semiconductor materials are crucial for accomplishing charge separation. Numerous studies have shown that coupling two semiconductor nanoparticles with differing band gap widths is one of the best strategies to lower electron-hole pair recombination and, as a result, increase photocatalytic activity. Additionally, there is more organic pollutant breakdown in these systems. In order to create visible-light photocatalysts, a number of narrow band gap metal oxides that provide novel ways to collect photons in the visible range have been combined. A variety of linked systems, including Cu2O-ZnO, have so far been shown to have some visible-light photocatalytic activity. For instance, in a coupled system of Cu2O and ZnO, Cu2O has a comparatively greater Conduction Band level than ZnO. As a result, the photogenerated electrons are moved to ZnO's CB, where they function as catalysts for a variety of reduction processes. This system is categorised as a "Type-A hetero junction," meaning that it may participate in the partial breakdown or reduction reaction of organic contaminants. However, since the OH radicals are not present on the ZnO surface, the evolution of CO2 that results from the total oxidation of organic contaminants would be sluggish.

II. LITERATURE REVIEW

On the other hand, visible light sensitisation might cause the hole-transfer from the sensitizer to the nanoparticle if the sensitizer's Valence Band level is lower than the nanoparticle's. As a consequence, holes may be created in the metal oxide nanoparticle's VB, which may then start a number of other oxidation processes. Given the strong oxidative potential of the holes in ZnO's VB, full and effective oxidation of organic molecules is anticipated in this system in the presence of light, such as Ag3PO4/ZnO heterojunction.

With a band gap of 2.45 eV, the semiconductor Ag3PO4 has garnered a lot of interest lately as a possible visible light photocatalyst. Its VB potential is lower than ZnO's, at 2.6 eV, and its Conduction Band and Valence Band edge potentials are 0.45 eV and 2.9 eV, respectively. In the Ag3PO4/ZnO system, where ZnO serves as a substrate and Ag3PO4 acts as a sensitizer by absorbing visible light, Ag3PO4 is thus thought to be a suitable sensitizer to enhance photocatalytic activity.

As an example, the ZnO/ZnS/CuS ternary nano photo catalyst under visible light has a higher photo catalytic activity than ZnO nanoparticles and ZnO/ZnS binary nano composite. The ternary nano composites also have high visible light photo catalytic activity and organic dyes can decompose efficiently. This is because the interface between the phases can serve as a separation site for photogenerated electron and holes due to the difference in energy levels of their valance band and conduction band. This reduces the recombination of photogenerated electron and holes and increases the photocatalytic activity of ZnO/ZnS/CuS under visible light.

Synthesis method of nano composites

The synthesis of nanomaterials may be done in two broad ways. both top-down and bottom-up methods. The term "top-down approach" describes the process of breaking a bulk substance into nanoparticles one by one. The term "bottom up approach" describes how a substance is produced from atoms or molecules. With a wide range of applications, the top-down and bottom-up methods may both be used in gas, liquid, or solid states. Controlling the size, shape, distribution, composition, and degree of aggregation of the particles is of importance to these applications. The next sections provide a quick overview of some of the well-known top-down and bottom-up synthesis techniques before going into detail on the synthesis and processes used in the current study.

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Top-down method

Using a top-down method, the bulk material is broken down into nanoscale structures or particles. Techniques for creating micron-sized particles have been expanded upon via top-down synthesis. These methods, which are simpler by nature, rely on either the division or removal of bulk material or the miniaturisation of bulk manufacturing procedures in order to create the necessary structure with the right attributes. The top-down method's primary drawback is the introduction of internal tension, surface flaws, and contaminants. For instance, lithography-produced nanowires have a rough surface and may have several contaminants and structural flaws. These methods include, but are not limited to, gas-phase condensation, atomic force manipulation, high-energy wet ball milling, electron beam lithography, and aerosol spraying.

Gas phase condensation: The easiest method for creating nanoparticles is to heat the target substance in a crucible that can withstand heat. Only materials with a high vapour pressure at hot temperatures—which may reach up to 2000°C—are suitable for this technique. Normal methods of introducing energy into the precursor include Joule heating, electron beam heating, and arc heating. The atoms evaporate into an atmosphere that might be reactive or inert. Reactive synthesis requires the feeding of materials with extremely low vapour pressure into the furnace in the form of a suitable precursor, such as organometallics, which break down to generate a condensable substance within the furnace. By colliding with the atoms of the cool gas, the hot atoms of the evaporated substance lose energy and condense into tiny clusters by homogeneous nucleation. When a product is being synthesised, these precursors combine with a substance that is separately delivered into the reaction chamber to generate the compound via a gas phase reaction. If the clusters stayed in the supersaturated zone, they would keep growing. They must be quickly extracted by a carrier gas from the supersaturated environment in order to regulate their size. There are only three factors that determine the size and distribution of the cluster: the rate of evaporation the rate of condensation the rate of gas flow (Figure 1).

ZnS nanoparticles were synthesised by an aqueous chemical process using sodium sulphide and zinc chloride as raw materials. Distilled water was used throughout the whole procedure due to its inherent benefits of simplicity and environmental friendliness. Every stage of the synthesis was carried out at low ambient temperature settings. A typical preparation included making a solution of 1 M zinc chloride in 100 mL of deionized water, adding 1 M sodium sulphide solution drop by drop to the mixture, then agitating the mixture with a magnetic stirrer at 70°C to generate a ZnS nanocolloid. Centrifugation at 2000 rpm for 15 minutes was used to gather the nanoparticles, and an ultrasonic bath was used to further purify them. After drying, the end product was used to break down dye by photocatalysis.

Sol-gel synthesis: The low-temperature chemical preparation of inorganic materials is known as sol-gel technology. A lot of researchers focused on creating novel photocatalysts. Nanoparticles have long been synthesised using sol-gel methods. A common method uses a metal precursor in an acidic or basic solution to form a sol. This process consists of three key steps: hydrolysis, condensation and growth. The metal precursor, to put it briefly, hydrolyzes in the medium, condenses to create a sol, and then polymerizes to form a network (Figure 2). This technique may be scaled up from laboratory to industrial levels due to its inherent simplicity.

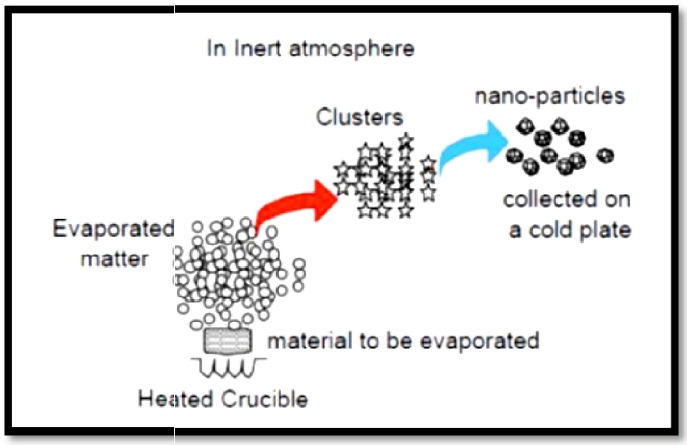

Figure 1: Representation of gas phase synthesis process of nanomaterial from a heated crucible.

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Bottom-up method

The "bottom-up" strategy is an option that may result in less waste and be more cost-effective. The accumulation of a substance from the bottom is referred to as a bottom-up approach: Cluster by cluster, molecule by molecule, or atom by atom. Many of these methods are either in the early stages of development or are just starting to be used in the commercial manufacturing of nanopowders. Luminescent nanoparticles may be created via a variety of well-known bottom-up procedures, including the organometallic chemical route, revere-micelle approach, sol-gel synthesis, colloidal precipitation, hydrothermal synthesis, template aided sol-gel, electrode position, and others. The primary benefit of colloidal precipitation is that water is employed as a solvent and non-toxic precursors are utilised.

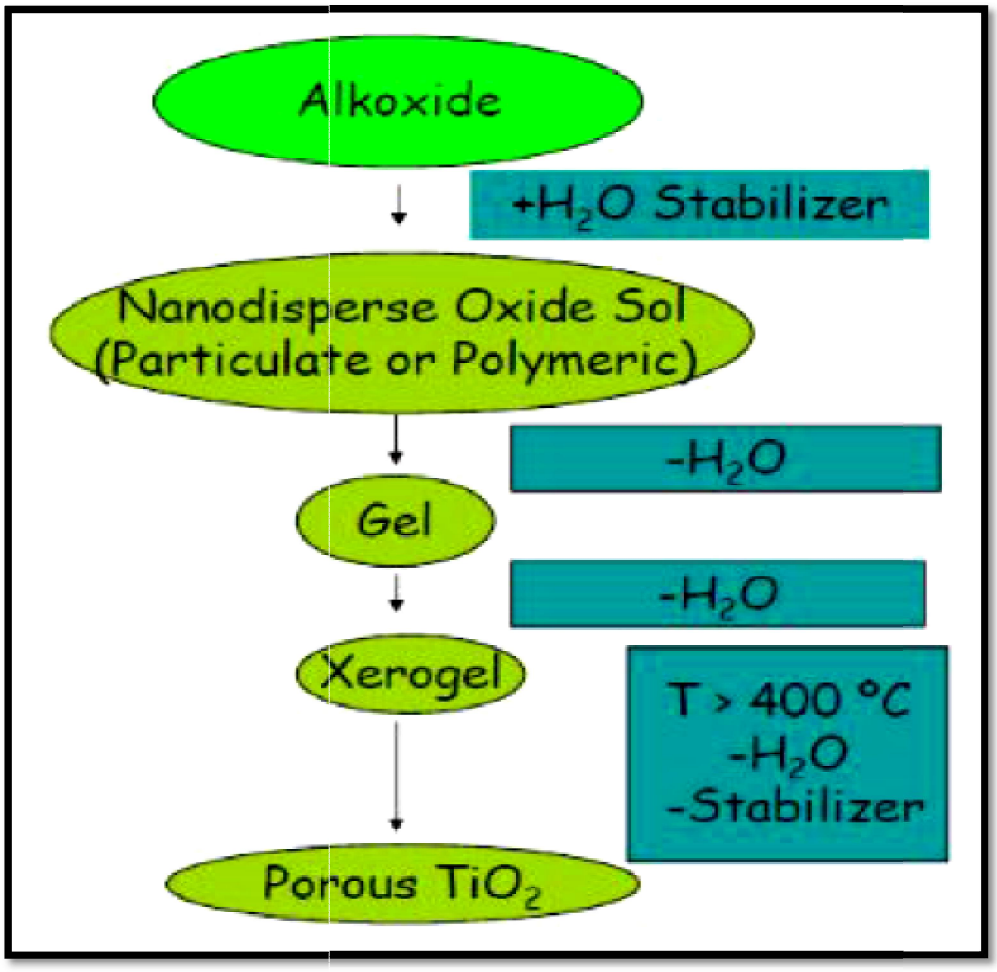

Figure 2: TiO_2 nanoparticle mediated mesoporous film preparation by sol-gel processing.

Discussion

CdS, ZnS, and PbS are examples of II-VI and IV-VI nanoparticles that have been created using this technique. For instance, ZnS nanoparticles have been created by combining alcohol-and sodium sulfide-based solutions of Zn acetate, then carefully ageing the mixture in the air. Furthermore, in order to determine its optical conductivity, a ternary composite of TiO2/ZrO2/SiO2 was synthesised using the sol-gel technique from Ti 4, Zr4, tetraethoxysilane Si4, and Acetylacetone as a complex form. Similar to the Zn2, SnCl4, and Phosphotungstic Acid hydrate solutions, the ternary SnO2-ZnO-ZnWO4 nanocomposite was also prepared via a sol-gel method for the purpose of assessing the sample's photocatalytic activity on the breakdown of 4-nitrophenol and the partial oxidation of 4-methoxybenzyl alcohol to p-anisaldehyde.

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The sol-gel approach has garnered the most interest among the other techniques because to its cheap process costs, ease of composition control, suitability for scaling up, and comparatively low calcination temperatures. Two of the sol-gel process's primary drawbacks are its wide size dispersion and high defect concentration. As a result, this synthesis method is seldom used.

Coprecipitation: Coprecipitation reactions occur when nucleation, growth, coarsening, and/or agglomeration activities take place all at once. The following features are seen in precipitation reactions: Most of the time, the results are intractable species that were created in very super saturated environments. One crucial stage is nucleation, which results in the formation of many tiny particles. Secondary processes including aggregation and Ostwald ripening have a significant impact on the products' shape, size, and characteristics. Usually, a chemical reaction produces the hyper saturation conditions required to cause precipitation. For instance, the carbonates of the metal salts Zn2.2H2O, Cu3.6H2O, and Ce23.4H2O and a solution of Na2CO3, which was used for the degradation of methyl violet dye and antibacterial activity, were co-precipitated to create the CeO2/CuO/ZnO metal ternary composite. Similar to this, NaOH, Co2.6H2O, and Ce3.6H2O solutions were used to create ternary composite oxide catalysts of CuO/Co3O4-CeO2 with a broad temperature window for the preferential oxidation of CO in H2 rich streams.

Hydrothermal synthesis: Because of its ease of use and high output, hydrothermal synthesis is an appealing technique. Furthermore, it results in the creation of nanoscale compounds under supercritical conditions that are not accessible by conventional methods. For the manufacture of inorganic nanomaterials such oxides, sulphides, phosphates, zeolites, and diamond, the hydrothermal process is a particularly efficient technique. This approach allows for good control over the morphology, phase homogeneity, and particle sizes and their distributions. The temperature and pressure within the reaction vessel determine how the precursors break down in a solvothermal or hydrothermal process at a given solvent.

For instance, AgNO3 and Na3PO4 solutions were used to create ZnO nanorods using a solvothermal technique. Moreover, Cu(NO3)2•3H2O, Al3•9H2O, Zn2•6H2O, and Polythene Glycol were used in conjunction with glucose-based carbonaceous materials as a template to manufacture the ZnO/CuO/ZnAl2O4 ternary composite.

Chemical bath deposition method: A sequential chemical bath deposition approach was used to carry out the electron deposition process. As an example, the TiO2/ZnO/CdS ternary hybrids were created using the electron deposition technique Curr Synthetic Sys Biol, Vol. 10 Iss. 6 No: 1000015. The photocatalytic activities of the TiO2/ZnO/CdS semiconductor composite were assessed by using the photodegradation of Alizarin Red S solution under UV photoirradiation. In these methods, TiO2/ZnO binary nanocomposites were employed as the starting material for the deposition of CdS thin films.

Microemulsion method: A collection of randomly placed droplets on a three-dimensional lattice that are capable of moving and colliding with one another is the representation of the microemulsion. Depending on the kind of nanoparticle, each simulation starts with the two or three sets of microemulsion droplets being randomly distributed: If the simulated nanoparticle is simple, the one-pot approach may be used to mimic it by combining equal volumes of two microemulsions with the reaction A+B→P, one containing reactant A and the other carrying reactant B.

Zinc oxide nanostructures with different morphologies were generated using the microemulsion technique. Three microemulsions are combined, one containing the metal salt A, the second containing the metal salt B, and the third containing the reducing agent R, to replicate the production of a bimetallic nanoparticle.

Incipient wetness impregnation method: The manufacture of heterogeneous catalysts is often accomplished using the intermittent wetness impregnation approach. The active metal precursors are usually dissolved in an organic or aqueous solution. Next, a catalyst support with the same pore volume as the volume of solution introduced is filled with the metal-containing solution. To deposit the metal on the catalyst surface, the catalyst may be dried and calcined in order to eliminate the volatile components present in the solution.

In-situ polymerization method: The inorganic nanoparticle is always synthesised in a polymer matrix using the in-situ polymerisation synthesis process, which occurs between the polymer monomer and the inorganic colloid in the presence of a strong oxidant. Inorganic nanoparticles are used to polymerise polymer monomers in the presence of a strong oxidant. Since polymerisation always begins at a temperature lower than 10°C, this preparation process does not need a high temperature. It's an easy and efficient way to make nanocomposites. Using appropriate precursors, this technique enables the one-step creation of nanocomposites including in situ produced nanoparticles. It is possible to

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grow the nanoparticles within the polymer matrix in this situation. This method has the benefit of maintaining a proper spatial distribution inside the polymer matrix while preventing particle agglomeration (Figure 3).

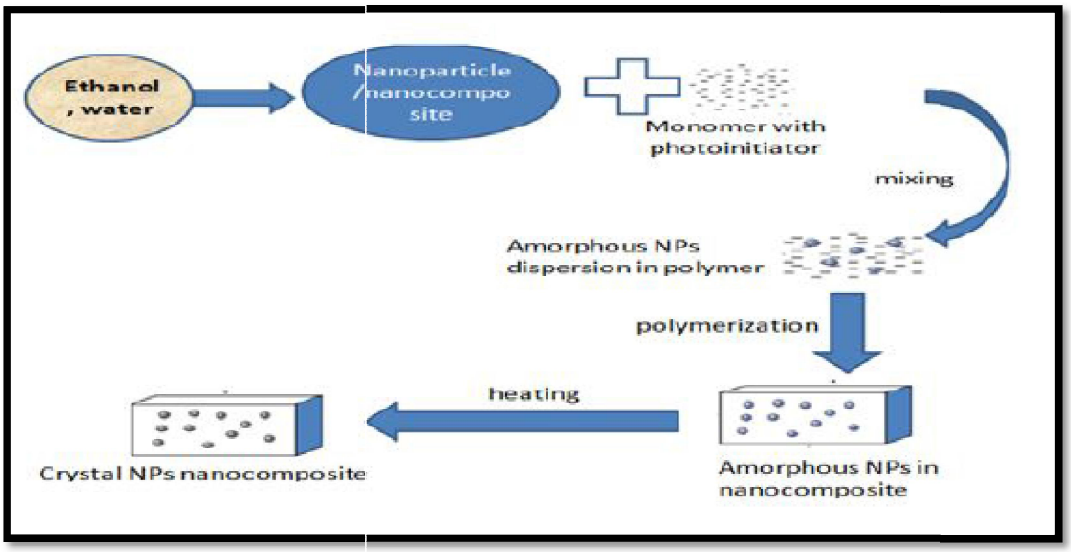

Figure 3: Schematic diagram for preparation routes of in-situ polymerization of nano composite in polymer matrix.

Approch to making semiconductors visible light active

In recent decades, semiconductor photocatalysis has been extensively researched for a broad range of applications, including water and air purification, hydrogen synthesis from water splitting, and more. However, broad band-gap semiconductors, which are only active under UV irradiation, make up the bulk of photo catalysts. Modifications must be made to the wide band-gap semiconductor photo catalyst in order to efficiently use visible sun light.

Many metal sulphides and oxides have been investigated as photocatalysts for the generation of hydrogen and environmental applications. However, most simple metal oxide photocatalysts are mainly active in the presence of UV irradiation, which is found in a limited fraction of solar light. For instance, the broad band-gap energies of 3.72 eV and 3.37 eV for ZnS and ZnO, respectively, hinder the use of visible light, which makes up the majority of solar energy. In recent times, noteworthy efforts have been undertaken to create novel or altered semiconductor photocatalysts that can use visible light. These endeavours include semiconductor coupling, metal ion doping, nonmetallic element doping, and organic dye sensitisation.

Band gap modification by doping

Nitrogen doping: With the help of visible light, electrons from ZnO's valence band undergo a two-step transition to the conduction band in a new band created by substitution N doping, which is located near to ZnO's VB. According to a density of states calculation, replacement nitrogen species produce states slightly above valence band maxima, where they may combine with O 2p valence states to reduce semiconductor band gaps. Recent studies suggest that using non-metal elements including N, F, S, P, and C may improve the desired band gap shrinking of ZnO. Due to band gap reduction, such modified ZnO demonstrated greater absorption in the visible region and improved organic pollutant degradation under visible light irradiation, particularly under solar light (Figure 4). The band gap narrows for Zn2+ ions with tetrahedral O coordination in the ZnO lattice due to sp-d exchange interactions between these transition metals' d electrons and conduction band electrons. Co and Cu doped ZnO has been shown to exhibit high visible light activity; as the Co content of ZnO grew, so did the absorption of visible light and the amount of surface oxygen vacancies. Because of its more superior crystalline structure and smaller band gap, Co doped ZnO photocatalysts outperformed Mn and Ni doped ZnO photocatalysts in terms of visible light activity.

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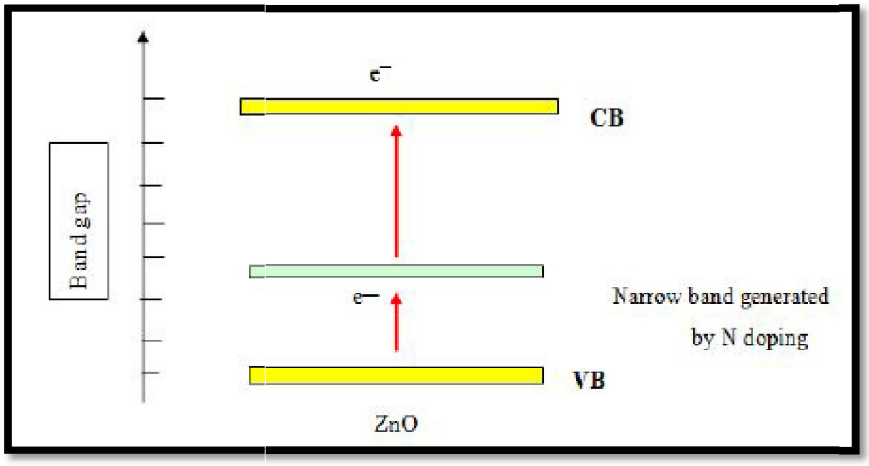

Figure 4: Visible light absorption by nitrogen doped ZnO.

Chromium doping: ZnS nanoparticles doped with chromium and containing 0.05, 0.1, 0.2, and 0.3 mol% of Cr were created by impregnation with incipient wetness. Under UV and visible light, the kinetics of the photocatalytic degradation of the dye Methyl Orange by synthesised nanoparticles was investigated. The effects of variables on the photocatalytic degradation of MO dye were examined, including dopant concentration, pH, and dye starting concentration. As the initial concentration of dye was increased, the rate of photocatalytic degradation reduced. After five hours of the process, the dye was destroyed by 74.28% and 65%, respectively, under visible and UV rays when 0.2 mol% Cr-ZnS was used as the photocatalyst. The red-shift of the Cr-doped ZnS photocatalyst's absorption edge, which allows it to collect more photons in the visible light, is thought to be the cause of the dye's higher photocatalytic degradation under visible light than under UV light.

Surface modification via organic materials and semiconductor coupling

Dye sensitization: Sensitising dye is also employed in semiconductors with larger band gaps to alter the mechanisms involved in the transfer of electrons during photocatalysis. An illustration of the photosensitization of a semiconductor is shown. The charge injection process is propelled by the energy differential between the conduction band of the semiconductor and the oxidation potential of the excited sensitizer.

Wide band gap semiconductors like ZnS and ZnO, which are otherwise inactive under visible light, can benefit from dye sensitisation as a useful tool to induce visible light photocatalysis on their surface. Physical dye adsorption takes place through a weak Vander Waals interaction between the dye molecule and the semiconductor surface. Electron transport between the dye molecules and the host semiconductor is facilitated by dye sensitisation.

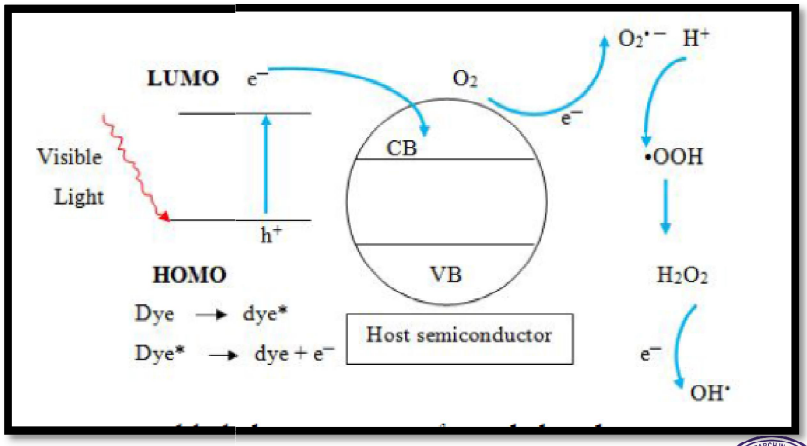

Figure 5: Visible light activation of a wide band gap semiconductor by dye sensitization.

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Catalytic activity in sensitised degradation is dependent on the energy of the generated electron and the degree of its separation. A straightforward method of inhibiting the back electron transfer is to create a long-range charge separation. It has been amply shown that by suppressing recombination, a greater distance between the electron and the oxidised dye improves catalytic effectiveness.

The following points sum up the essential conditions that a dye molecule has to meet in order to be taken into consideration as a suitable candidate for spectrum sensitisation of semiconductors. robust absorption throughout the whole visible spectrum. robust adherence to the semiconductor surface. Energy levels in the right places. quick electron transport to the ZnS as opposed to the dye's ground state degradation. stability after extended exposure to sunshine. inexpensive, straightforward, and repeatable purification and synthesis. To reduce energy losses, excited and ground state electron transport should have a small reorganisation energy.

Semiconductor coupling: According to Rajesh et al., zinc acetate, cadmium acetate, and hydrogen sulphide were used as the raw ingredients to create ZnS-CdS photocatalyst. Zn and Cd salts of known weight were used to make the semiconductor, which was then precipitated by running H2S through it. After allowing the precipitate to settle, additional H2S was added to the supernatant solution to confirm the precipitate's continued existence. After that, the precipitate was dried at room temperature and repeatedly cleaned with distilled water. Under visible light radiation, the photocatalytic activity of the as-synthesised photocatalyst was investigated for the photodegradation of crystal violet at various parametric effects. It was shown that at the ideal pH and starting ye concentration, the right quantity of ZnS-CdS catalyst could effectively decompose crystal violet (Figure 6).

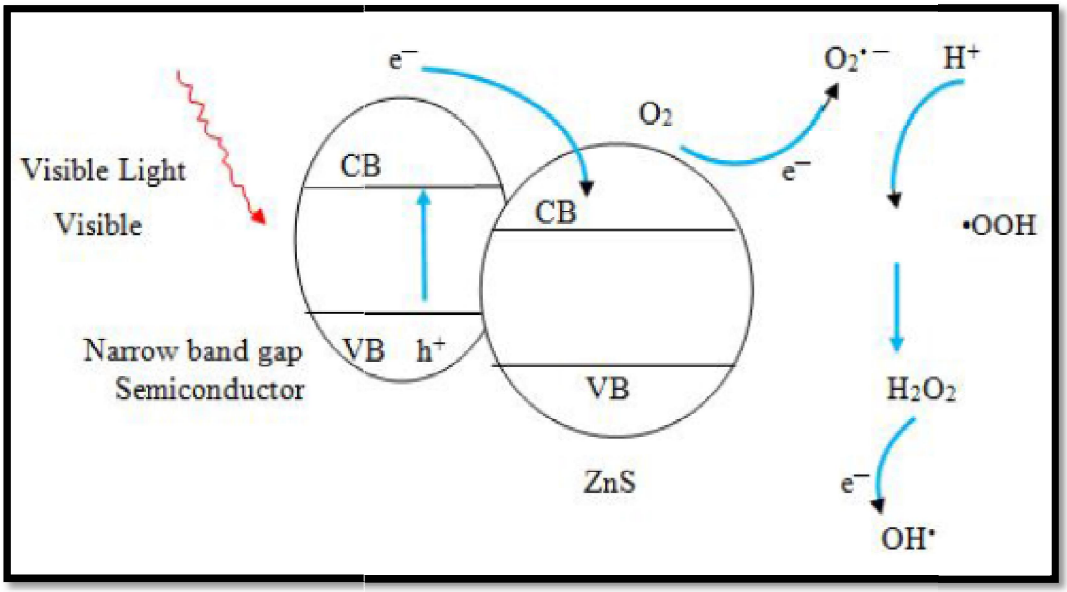

Figure 6: Visible light activation of ZnS by coupling with a narrow band gap semiconductor.

Recent research has also attempted to enhance the wide band gap photocatalysts' photocatalytic activity by coupling with other metal oxide systems. The band gap energy of the ternary composite linked system Fe2O3-ZnO-MnO2 is 2.53 eV. Compared to the 3.37 eV band gap energy of ZnO, this band gap energy is less. Combining semiconductor nanoparticles with band gap widths of ZnO and Fe2O3 results in this lowering band gap energy. One of the best uses to lessen the recombination of electron-hole pairs is semiconductor coupling. This is due to the possibility of hole transfer from the VB of high band gap semiconductor to the VB of low band gap semiconductor and CB electron ejection from the low band gap semiconductor to the band gap semiconductor upon exposure to visible light striking the VB of combined semiconductor. Furthermore, the excited electrons may be scavenged by the electron accepter Mn, which would completely stop electron-hole pair recombination and carrier charge separation. The ternary nano composites' strong photo catalytic activity in visible light and their ability to degrade organic dyes quickly suggest that they have a greater photo catalytic activity overall.

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III. CONCLUSION

The potential for mineralizing non-biodegradable contaminants in air and water is enormous with heterogeneous photocatalysis. Only a tiny percentage of solar light contains UV irradiation, which is the primary source of activity for most simple metal oxide photocatalysts. In recent times, noteworthy efforts have been undertaken to create novel or altered semiconductor photocatalysts that can use visible light. These endeavours include semiconductor coupling, metal ion doping, nonmetallic element doping, and organic dye sensitisation.

Numerous studies have shown that coupling two semiconductor nanoparticles with differing band gap widths is one of the best strategies to lower electron-hole pair recombination and, as a result, increase photocatalytic activity. Numerous metal oxides with tiny band gaps are opening up new possibilities for photon harvesting in the visible spectrum. Many linked system compounds have so far shown noticeably more photocatalytic activity than pure nano composites, as the coupling of nano composites encouraged the separation of holes and electrons. The ternary nano composites breakdown organic dyes effectively and exhibit strong visible light photocatalytic activity, suggesting that they have enhanced photocatalytic activity.

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IJARSCT
ISSN (Online) 2581-9429
International Journal of Advanced Research in Science, Communication and Technology (IJARSCT)
International Open-Access, Double-Blind, Peer-Reviewed, Refereed, Multidisciplinary Online Journal
Impact Factor: 7.53
Volume 4, Issue 2, September 2024

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