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REVIEW: SYNTHESIS, CHARACTERIZATION OF NANOMATERIALS AND THEIR APPLICATION IN PHOTOCATALYSIS

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ABSTRACT:

Present review emphatically introduces the synthesis, characterization and application of nanomaterial's including their advantages and disadvantages of various characterization techniques. The recent past in the technological development evidenced that evolution in Nanomaterials is the key factor. The present review article highlighted the types of nanoparticles and their synthesis methods, characterization techniques. There are many techniques and applications are reported in the last decade but here we focused on the general synthetic approaches, characterization techniques and the application as photocatalyst using nanomaterials which provide a general idea to the young researchers.
Keywords: Nanomaterials, Characterization, Synthesis technique, Green synthesis method, Biodiesel

INTRODUCTION

Photocatalysis is promising technology for the removal of organic pollutants from water and air. Photocatalysis is a process that uses light energy to activate a substance (called a photocatalyst) that can accelerate a chemical reaction. In the context of environmental remediation, photocatalysis can be used to degrade organic pollutants in water or air by using nanomaterials as photocatalysts. Nanomaterials have at least one dimension in the range of 1-100 nm. Some of the common nanomaterials used as photocatalysts are titanium dioxide (TiO_2), zinc oxide (ZnO), metal sulfides, and composite materials. They have unique properties such as large surface area, high reactivity, and tunable band gap that make them suitable for photocatalysis (1-5).

There are different types of nanomaterials that can be used for photocatalysis, depending on their composition, structure, shape, and functionality. Most common are further:

Semiconductor nanomaterials: These are nanomaterials that have a band gap between their valence and conduction bands, which allows them to absorb light and generate electron-hole pairs. The electron-hole pairs can then participate in redox reactions with the reactants or the

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catalyst surface. Examples of semiconductor nanomaterials include ZnO, TiO_2, Fe_2O_3, WO_3, and $SrTiO_3$ (06-07).

Metal nanomaterials: These are nanomaterials that consist of metallic elements or alloys, which have high electrical conductivity and can act as co-catalysts or electron mediators for semiconductor nanomaterials. Metal nanomaterials can also exhibit plasmonic effects, which enhance the light absorption and scattering of the photocatalysts. Examples of metal nanomaterials include Au, Ag, Pt, Pd, and Cu.(8-10).

Carbon-based nanomaterials: These are nanomaterials that consist of carbon atoms arranged in various forms, such as graphene, carbon nanotubes, carbon dots, fullerenes, and graphitic carbon nitride. Carbon-based nanomaterials can have high stability, conductivity, and surface functionality, which make them suitable for photocatalysis applications. Carbon-based nanomaterials can also act as supports or modifiers for other nanomaterials. (11)

Composite nanomaterials: These are nanomaterials that consist of two or more types of materials that are physically or chemically combined to form a heterostructure. Composite nanomaterials can improve the photocatalytic performance by enhancing the light absorption, charge separation, charge transfer, and catalytic activity of the individual components. Examples of composite nanomaterials include core-shell structures, solid solutions, type-II heterojunctions, and Z-scheme systems.(12-14)

These are some of the types of nanomaterials that can be used for photocatalysis. However, there are many other factors that can affect the photocatalytic performance of these nanomaterials, such as size, shape, surface area, surface defects, doping, coating, and coupling. Therefore, the design and optimization of these nanomaterials require careful consideration of their synthesis methods, characterization techniques, and application conditions

- **Methods of synthesis of nanomaterial :-**

There are many **methods of synthesis of nanom**aterials used in photocatalysis, which can be classified into two major types: top-down and bottom-up. The top-down methods involve breaking down the bulk material into smaller particles by physical or mechanical techniques, such as milling, grinding, etching, or lithography. The bottom-up methods involve building up the nanoparticles from atoms or molecules by chemical or biological processes, such as precipitation, sol-gel, hydrothermal, microwave, Sono chemical, or biosynthesis.(15-19)

Some examples of **top-down** methods are (20-21):-

i) Ball milling: This method uses a rotating device to grind the bulk material into fine powder under high-energy impact. The size and shape of the nanoparticles can be controlled by adjusting the milling time, speed, and ball-to-powder ratio. Nanomaterials like ZnO ,TiO_2, and Fe_2O_3 are widely used.

ii) Laser ablation: This method uses a high-intensity laser beam to vaporize the target material in a liquid or gas medium. The vaporized atoms or molecules then condense into nanoparticles in the surrounding medium. The size and shape of the nanoparticles can be controlled by adjusting the laser power, wavelength, pulse duration, and medium composition. For example Au, Ag and CuO with high photocatalytic activity.

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iii) Lithography Technique: It is a key technique in the synthesis and fabrication of nanomaterials, particularly in the field of nanotechnology and semiconductor manufacturing. It involves using patterns to create very small features on a substrate, typically a silicon wafer, to build up structures at the nanoscale. Here are some common lithographic techniques used in nanomaterial synthesis. Photolithography uses light to transfer a geometric pattern from a photomask to a light-sensitive chemical photoresist on the substrate. The areas exposed to light are chemically altered, allowing selective removal to create the desired patterns.

X-ray Lithography uses X-Lrays to transfer a pattern from a mask to a resist-coated substrate. The shorter wavelength of X-rays allows for higher resolution compared to photolithography.

iv) Etching: It is techniques are crucial for synthesizing nanomaterials, especially in microelectronics, nanofabrication, and materials science. Etching involves removing material from a substrate to create nanostructures with specific patterns or features. There are two primary types of etching: **wet etching** and **dry etching**. Each type has several methods, which are used depending on the material and desired precision.

v) Mechanical Cutting vi) Laser Interference Ethography vii)Ion Implantation

These all are crucial techniques used as per the requirement physical and chemical approach of nanomaterials.

Bottom-Up Approach are as follows (22-23):-

i) Sol-gel: This method uses a chemical reaction between a metal precursor (such as a metal salt or alkoxide) and a solvent (such as water or alcohol) to form a gel-like network of metal oxide nanoparticles. The gel can then be dried and calcined to obtain the final product. The size and shape of the nanoparticles can be controlled by adjusting the precursor concentration, pH, temperature, and drying conditions. This method can produce various metal oxide nanoparticles, such as TiO_2, ZrO_2, and SnO_2, which have excellent photocatalytic performance. The sol-gel method is a wet-chemical technique that is widely used for the synthesis of nanomaterials, especially metal oxide nanoparticles (24-29). It involves the following steps:

A metal precursor, such as a metal salt or alkoxide, is dissolved in a solvent, such as water or alcohol, to form a sol. A sol is a colloidal solution of nanoparticles dispersed in the solvent.

The sol is then converted into a gel by heating, stirring, or adding a catalyst. A gel is a solid network of nanoparticles interconnected by chemical bonds, with the solvent trapped in the pores.The gel is then dried and calcined to remove the solvent and obtain the final product. The drying and calcining conditions can affect the size, shape, and structure of the nanoparticles.

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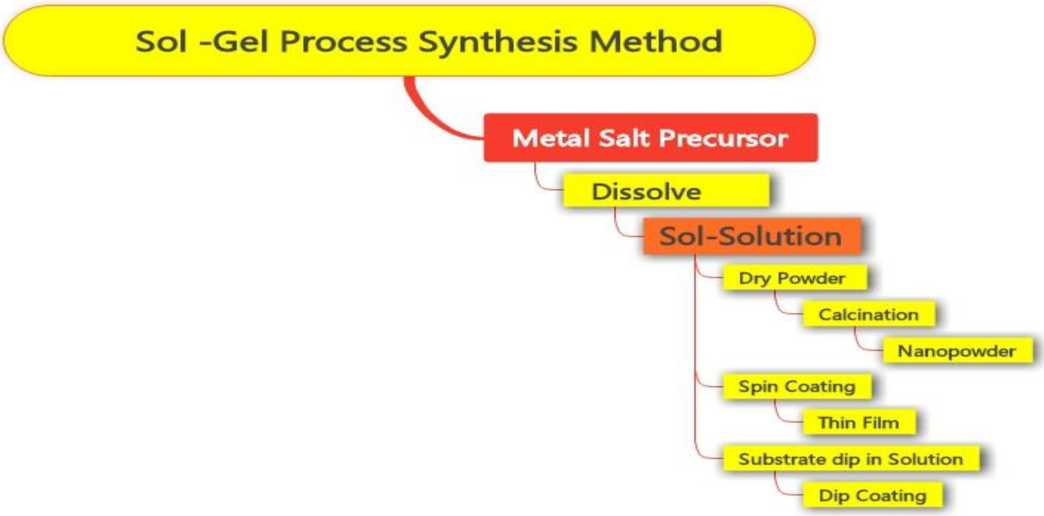

Fig:1. Diagram showing Sol-Gel Synthesis Method

The sol-gel method has many advantages over other methods.

ii. Chemical precipitation method

It is a technique for preparing nanoparticles by using a chemical reaction to form solid particles from a solution (30).

iii. Hydrothermal method

The hydrothermal method is a technique that uses a high-pressure and high-temperature aqueous solution to dissolve the metal precursor and then precipitate the metal oxide nanoparticles. The solution is usually sealed in an autoclave and heated above the boiling point of water. The size and shape of the nanoparticles can be controlled by adjusting the precursor concentration, pH, temperature, pressure, and reaction time. This method can produce various metal oxide nanoparticles with different morphologies, such as nanorods, nanosheets, and nanoflowers, which have enhanced photocatalytic activity (31-33).

TiO_2 -SiO_2 nanoparticles, which are prepared by adding titanium iso propoxide to a solution of SiO_2 nanoparticles. These nanoparticles have enhanced photocatalytic activity and stability compared to pure TiO_2 nanoparticles.

iv. Chemical Vapor Deposition (CVD) is a widely used method for the preparation of nanomaterials, including thin films, nanoparticles, nanowires, and more. It involves the controlled deposition of materials from vapor-phase precursors onto a substrate(34-38). Here's a general overview of the CVD method for preparing nanomaterials:

v. Physical Vapor Deposition (PVD) is a widely used method for the preparation of nanomaterials, especially thin films and coatings. PVD relies on physical processes to deposit nanoscale materials onto a substrate(39-40). Here's an overview of the PVD process for nanomaterial preparation:

vi. Electro-chemical synthesis is a versatile and widely used method for the preparation of nanomaterials. This approach leverages electrochemical reactions to control the growth and

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deposition of nanoscale materials(41-42). Here's an overview of the electrochemical synthesis process for nanomaterials:

vii. Template-assisted synthesis is a versatile and widely used method for the preparation of nanomaterials with controlled size, shape, and structure. This approach relies on using templates as molds or scaffolds to guide the growth or assembly of nanomaterials(43).

viii. Green synthesis of nanomaterials refers to the environmentally friendly and sustainable methods used to produce nanomaterials while minimizing the use of hazardous chemicals and energy consumption. This approach is becoming increasingly important due to its potential to reduce the environmental impact of nanomaterial production(44-49).

ix. Electrospinning is a versatile and widely used method for the preparation of nanomaterials in the form of nanofibers and nanofibrous mats. This technique relies on an electric field to draw and stretch a polymer or precursor solution into ultrafine fibers with diameters in the nanometer range (50).

Characterization techniques : -

Characterization techniques for nanomaterials are essential tools that allow researchers to understand and analyze the properties, structure, and behavior of nanoscale materials. These techniques provide valuable information about the size, shape, composition, surface characteristics, and other key attributes of nano materials (51). Here are some commonly used characterization techniques for nanomaterials:

Scanning Electron Microscopy (SEM): SEM uses a focused beam of electrons to scan the surface of nanomaterials, providing high-resolution images and information about the topography, morphology, and size of nanoparticles.

Transmission Electron Microscopy (TEM): TEM uses transmitted electrons to obtain extremely detailed images of nanomaterials at the atomic or nanometer scale. It can reveal information about crystal structure, lattice defects, and particle size distribution.

Atomic Force Microscopy (AFM): AFM measures forces between a sharp tip and the nanomaterial's surface, producing high-resolution images and providing information about surface roughness and mechanical properties.

X-ray Diffraction (XRD): XRD is used to determine the crystal structure of nanomaterials by analyzing the diffraction patterns of X-rays when they interact with the material's atomic lattice. This technique is vital for identifying phases and crystallinity.

Dynamic Light Scattering (DLS): DLS measures the Brownian motion of nanoparticles in a liquid to determine their hydrodynamic size and size distribution. It is commonly used for nanoparticles in solution.

Fourier Transform Infrared Spectroscopy (FTIR): FTIR analyzes the interaction of infrared radiation with nanomaterials, providing information about chemical composition and functional groups on the surface.

Raman Spectroscopy: Raman spectroscopy measures the inelastic scattering of photons by nanomaterials, yielding information about molecular vibrations and crystal structures. It can be used to identify chemical compounds and detect defects.

X-ray Photoelectron Spectroscopy (XPS): XPS analyzes the elemental composition and chemical state of the nanomaterial's surface by measuring the energy of emitted photoelectrons.

Nuclear Magnetic Resonance (NMR) Spectroscopy: NMR provides insights into the atomic and molecular structures of nanomaterials by studying the nuclear magnetic properties of atoms.

UV-Visible Spectroscopy: UV-Visible spectroscopy measures the absorption of ultraviolet and visible light by nanomaterials, providing information about their electronic structure and optical properties.

Surface Area Analysis (BET): Brunauer-Emmett-Teller (BET) analysis is used to determine the specific surface area of nanomaterials, which is critical for understanding their reactivity and adsorption properties.

Thermogravimetric Analysis (TGA): TGA measures the change in mass of nanomaterials as a function of temperature, allowing the determination of thermal stability, decomposition, and adsorbed water content.

- **General mechanism of photocatalytic degradation of organic pollutants**

Photocatalysis is a process that uses light and a catalyst to accelerate a chemical reaction. The photocatalytic degradation of organic pollutants is a complex process that involves the use of a photocatalyst to break down organic compounds into simpler, less harmful substances in the presence of light. (1-5)

General mechanism of photocatalytic degradation of organic pollutants using nanomaterials involves following steps.

1. **Mass transfer**: The organic pollutant molecules diffuse from the bulk solution to the surface of the nanomaterial.

2. **Adsorption:** The organic pollutant molecules adsorb on the surface of the nanomaterial, forming a thin layer.

3. **Excitation:** The nanomaterial absorbs light energy and generates electron-hole pairs in its conduction and valence bands, respectively

The process begins with the absorption of photons (light) by the photocatalyst material. Photocatalysts are typically semiconductors, such as titanium dioxide (TiO_2) or zinc oxide (ZnO) , that have a bandgap energy corresponding to the energy of the incoming photons. When a photon with sufficient energy is absorbed by the photocatalyst, it promotes an electron from the valence band to the conduction band, creating an electron-hole pair.

4. **Generation of Electron-Hole Pairs:** The absorbed photon excites an electron from the valence band (the electron's lower-energy state) to the conduction band (the electron's higher-energy state). This process leaves behind a positively charged hole (a "hole" in the valence band), which is also called a "positive electron vacancy."

5. **Migration of Electrons and Holes:** The photogenerated electrons and holes are mobile within the semiconductor material. Electrons migrate to the surface of the photocatalyst, while holes migrate away from the surface into the bulk of the material. This separation of charges is critical for the subsequent redox reactions.

6.**Redox Reactions**: Once at the surface, the photogenerated electrons and holes can participate in redox (reduction-oxidation) reactions with adsorbed organic pollutants. There are two main pathways for these reactions:

a. **Direct Pathway**: In this pathway, the photogenerated electrons (e^-) in the conduction band can directly react with adsorbed organic molecules, donating electrons to reduce them. Meanwhile, the holes (h+) in the valence band can oxidize water or other species present on the photocatalyst surface. The resulting reactive species, such as hydroxyl radicals (•OH), superoxide radicals ($•O^{2-}$), and peroxide species ($•O2^{-2}$), play a crucial role in the degradation of organic pollutants. These highly reactive species attack and break down the pollutant molecules into simpler and less harmful compounds.

b**. Indirect Pathway**: In the indirect pathway, the photogenerated holes (h+) in the valence band react with water or adsorbed oxygen molecules to form reactive oxygen species (ROS), such as hydroxyl radicals (•OH). These ROS then participate in the oxidation of organic pollutants.

7. **Complete Degradation**: The series of redox reactions initiated by the photogenerated electrons and holes continue until the organic pollutant molecules are completely mineralized into carbon dioxide (CO_2), water (H_2O), and other innocuous byproducts. This mineralization process ensures the complete removal of the organic pollutant from the environment.

8. **Desorption:** The degraded products desorb from the surface of the nanomaterial and return to the bulk solution.

➢ **Enhanced photocatalytic activity**

Enhanced photocatalytic activity is a term that refers to the improvement of the efficiency and selectivity of a photocatalytic process **.**The photocatalytic degradation of organic pollutants depends on several factors, such as the type and concentration of the pollutant, the pH, the presence of natural organic matter, the adsorption of the pollutant on the catalyst surface, the light intensity and wavelength, the catalyst loading and morphology, and the presence of co-catalysts or inhibitors. The mechanism of photocatalytic degradation also varies depending on the structure and functional groups of the organic pollutant. Some pollutants can be directly oxidized by the holes or electrons on the catalyst surface, while others need to be activated by ROS in the bulk solution. The degradation products and pathways can be identified by using analytical techniques, such as chromatography and mass spectrometry

Photocatalytic degradation is a promising technology for the treatment of water contaminated by organic pollutants, such as pesticides, pharmaceuticals, dyes, cyanotoxins, and taste and odor compounds. It has several advantages over conventional methods, such as low cost, high efficiency, environmental friendliness, and possibility of solar energy utilization. However, there are also some challenges and limitations that need to be overcome, such as low quantum yield, catalyst deactivation, incomplete mineralization, formation of toxic intermediates, and energy consumption.

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