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A STUDY ON NANOCOMPOSITES OF ZINC IRON OXIDE SYNTHESIS AND CHARACTERIZATION

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Abstract

Nanocomposites are materials that combine two or more constituent materials at the nanoscale, resulting in a new material with enhanced properties. Zinc iron oxide (ZnFe2O4) nanocomposites are particularly interesting due to their unique magnetic, optical, and catalytic properties. These properties are a result of the synergy between the zinc ferrite matrix and the incorporated nanoparticles or nanofillers. The article will explore various synthesis methods, characterization techniques, and the potential applications of these materials. The properties of zinc iron oxide nanocomposites are highly dependent on their synthesis method, which dictates their size, shape, and purity. Co-precipitation method is a common and cost-effective technique. It involves precipitating zinc and iron ions simultaneously from a solution using a base. The reaction parameters like pH, temperature, and stirring speed are crucial for controlling the final product's morphology. The Hydrothermal/Solvothermal method involves a chemical reaction in a sealed vessel at high pressure and temperature. It allows for better control over the crystallinity and morphology of the nanoparticles. The solvothermal method uses an organic solvent instead of water. Sol-gel method involves creating a colloidal suspension (sol) of precursors, which then forms a gel network. Subsequent drying and calcination lead to the formation of the nanocomposite. This method is known for producing highly homogeneous materials with excellent purity. Combustion method is a rapid and energy-efficient method where a mixture of metal nitrates and a fuel (like citric acid or urea) is ignited. The combustion reaction produces a fluffy powder of the nanocomposite.

Keywords:

Nanocomposites, Zinc, Iron, Oxide, Characterization

Introduction

To understand the properties and structure of the synthesized nanocomposites, several characterization techniques are employed. X-ray Diffraction (XRD) is used to determine the crystal structure, phase purity, and crystallite size of the nanocomposite. The diffraction pattern provides information on the lattice parameters and the presence of any impurities. (Wojnarowicz, 2020)

Scanning Electron Microscopy (SEM) and Transmission Electron Microscopy (TEM) provides a high-resolution image of the surface morphology and particle size distribution. TEM, on the other hand, gives a detailed view of the internal structure, shape, and size of individual nanoparticles.

Vibrating Sample Magnetometer (VSM) is used to measure the magnetic properties, such as saturation magnetization (Ms), coercivity (Hc), and remanence (Mr). Zinc ferrite is a soft magnetic material, and its nanocomposite form can exhibit superparamagnetic behavior, which is size-dependent.

UV-Vis Spectroscopy technique is used to study the optical properties, specifically the band gap of the material. The band gap can be calculated from the absorption spectrum using the Tauc plot. The nanocomposite's band gap is often different from that of its individual components.

Fourier Transform Infrared (FTIR) Spectroscopy is used to identify the chemical bonds and functional groups present in the material. It helps to confirm the formation of the zinc iron oxide structure. Thermogravimetric Analysis (TGA) is used to study the thermal stability of the nanocomposite. It measures the change in weight as a function of temperature. (Mutukwa, 2022)

The co-precipitation method is a widely used and effective wet-chemical technique for synthesizing nanocrystalline materials, including zinc-iron oxide nanocomposites. This method is preferred for its simplicity, cost-effectiveness, and ability to produce

materials with high crystallinity and uniform particle size. The basic principle involves simultaneously precipitating two or more metal precursors from a homogeneous solution by adjusting the pH.

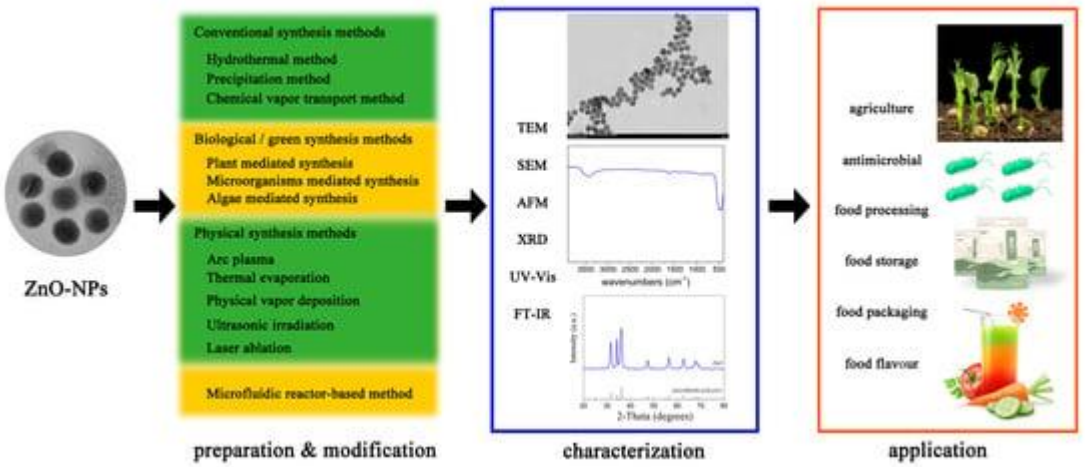

Figure 1 : Nanocomposites of Zinc Iron Oxide Characterization

To synthesize zinc-iron oxide nanocomposites, the primary precursors are typically a zinc salt (such as zinc chloride or zinc sulfate) and an iron salt (such as ferric chloride or ferrous sulfate). An alkaline solution, most commonly sodium hydroxide (NaOH) or ammonium hydroxide (NH4OH), is used as the precipitating agent. The choice of precursors and the precipitating agent can influence the final product's morphology and properties. Deionized water is used as the solvent to prevent unwanted impurities from entering the reaction. (Zhang, 2022)

The synthesis process generally follows these key steps:

Solution Preparation: Stoichiometric amounts of the zinc and iron precursors are dissolved in deionized water to create a homogeneous solution. The metal cations (Zn2+ and Fe3+) are dispersed uniformly throughout the solution.

pH Adjustment: A precipitating agent, like NaOH, is slowly added to the precursor solution while stirring vigorously. This increases the pH of the solution. The critical part of this step is to carefully control the pH, as this determines the precipitation of the metal hydroxides. The ideal pH range for co-precipitating zinc and iron hydroxides is typically between 10 and 12. Maintaining a constant pH is crucial to ensure that both metal ions precipitate simultaneously and in the correct ratio.

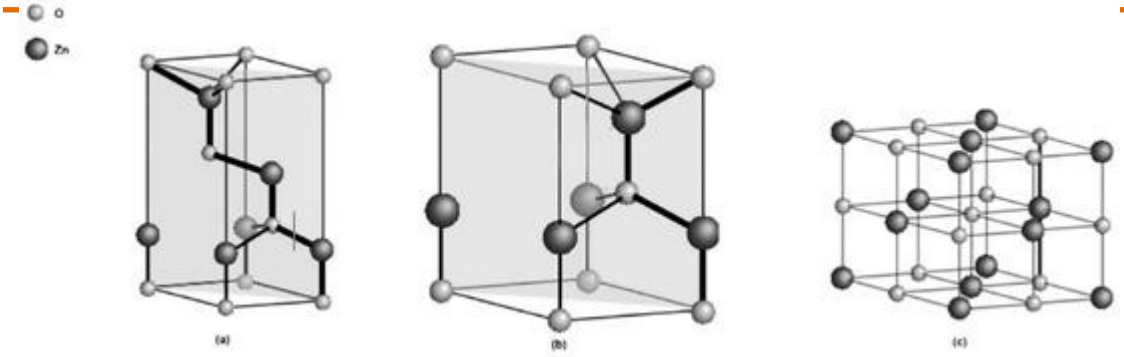

Figure 2. Crystal structure models of ZnO (**a**) zinc blende (**b**) wurtzite and (**c**) rock salt

Precipitation and Aging: As the pH rises, the zinc and iron ions react with the hydroxide ions (OH^-) to form their respective hydroxides, which are insoluble in the solution. This leads to the formation of a precipitate. The chemical reactions can be simplified as:

- $Zn^{2+} + 2OH^- \rightarrow Zn(OH)_2$
- $Fe^{3+} + 3OH^- \rightarrow Fe(OH)_3$

The resulting solution is often "aged" or allowed to sit for a period of time. This aging process promotes the growth of the nanoparticles and helps achieve a more uniform particle size distribution.

Washing and Filtration: The precipitated product is then separated from the solution using filtration or centrifugation. The precipitate is washed multiple times with deionized water and/or ethanol to remove any unreacted precursors, excess base, or by-products. This step is critical for obtaining a high-purity product.

Drying and Calcination: The washed precipitate is dried, typically in an oven at a relatively low temperature (around 80−100 °C), to remove residual moisture. Following this, the dried powder is subjected to calcination at a higher temperature (e.g., 400−600 °C). Calcination is a heat treatment process that converts the metal hydroxides into their corresponding oxides by driving off the water molecules. This final step forms the desired zinc-iron oxide nanocomposite.

After synthesis, the zinc-iron oxide nanocomposites are characterized using various analytical techniques to confirm their structure, composition, and properties. X-ray

diffraction (XRD) is used to determine the crystal structure and size. Scanning electron microscopy (SEM) and transmission electron microscopy (TEM) are employed to visualize the morphology and size of the nanoparticles. Techniques like energy-dispersive X-ray spectroscopy (EDX) and Fourier-transform infrared spectroscopy (FTIR) help confirm the elemental composition and chemical bonding.

These nanocomposites have numerous applications, particularly in fields like catalysis, biomedicine (e.g., drug delivery and hyperthermia), and electronics. Their magnetic and semiconducting properties make them suitable for use as gas sensors, photocatalysts, and in magnetic storage devices. The co-precipitation method, despite its challenges, remains a cornerstone for the synthesis of these advanced materials. (Lee, 2021)

Literature Review

Bokhari et al. (2022): The characterization process involves dispersing the zinc iron oxide nanocomposite powder in a solvent (or pressing it into a thin film) and placing it in a UV-Vis spectrophotometer. The instrument then measures the transmittance or absorbance of the sample across a range of wavelengths, typically from 200 nm to 800 nm. The raw data, usually in the form of absorbance vs. wavelength, is then converted into a plot of absorption coefficient vs. photon energy. Finally, a Tauc plot is constructed to determine the bandgap energy.

Shaba et al. (2021): UV-Vis spectroscopy is an indispensable technique for the optical characterization of zinc iron oxide nanocomposites. It provides critical information about the material's electronic structure and bandgap energy, which are fundamental to its potential applications in various fields. The data obtained from UV-Vis analysis, often complemented by other techniques like XRD and SEM, enables a comprehensive understanding of the nanocomposite's properties and performance.

Singh et al. (2021): The XRD pattern of a zinc iron oxide nanocomposite is a unique fingerprint that provides a wealth of information about its structure. The most fundamental application of XRD is identifying the crystalline phases present in the nanocomposite.

Hong et al. (2020): By comparing the positions of the diffraction peaks (2θ values) with established reference databases, like the JCPDS (Joint Committee on Powder Diffraction Standards) card files, it's possible to confirm the presence of phases such as zinc oxide (ZnO), iron oxides (Fe_2O_3 or Fe_3O_4), and zinc ferrite ($ZnFe_2O_4$). The presence of multiple distinct sets of peaks indicates a multiphase nanocomposite, while the absence of extra peaks suggests a high degree of phase purity. Any minor, unidentified peaks could signify impurities or secondary phases.

Prabha et al. (2021): The width of the diffraction peaks in an XRD pattern is inversely related to the size of the crystallites. Broad peaks indicate smaller crystallite sizes, which are characteristic of nanomaterials. This relationship is quantified by the Scherrer equation: $D=\beta\cos\theta K\lambda$ where D is the average crystallite size, K is a shape factor (typically ~0.9), λ is the X-ray wavelength, β is the full width at half maximum (FWHM) of the diffraction peak in radians, and θ is the Bragg angle. Besides crystallite size, peak broadening can also be caused by microstrain within the crystal lattice. Distinguishing between these two effects often requires more advanced analysis methods like the Williamson-Hall plot.

Agnieszka et al. (2021): The precise location of the diffraction peaks can be used to calculate the lattice parameters of the crystalline phases. For instance, zinc ferrite ($ZnFe_2O_4$) typically has a cubic spinel structure. By using the Miller indices (hkl) of the peaks and their corresponding 2θ values, the lattice parameter (a) can be calculated.

Sirelkhatim et al. (2020): Small shifts in the peak positions may indicate a change in the lattice parameters, which can be caused by factors like doping, defects, or changes in the chemical composition of the material. For example, in a $ZnO-Fe_2O_3$ nanocomposite, the peaks of the individual components might be slightly shifted compared to pure samples due to interfacial strain or alloying.

Nanocomposites of Zinc Iron Oxide Synthesis and Characterization

The hydrothermal and solvothermal methods are a class of wet-chemical techniques used to synthesize a wide range of materials, including inorganic compounds, metals, and nanomaterials. The core principle involves conducting a chemical reaction in a sealed vessel, or autoclave, at a temperature above the boiling point of

the solvent and under high pressure. When the solvent is water, the method is called hydrothermal. When a non-aqueous solvent is used (such as ethanol or toluene), it's termed solvothermal. These high-pressure, high-temperature conditions significantly increase the reactivity of the precursors and allow for the formation of crystalline materials that would be difficult or impossible to synthesize under ambient conditions.

The synthesis of zinc iron oxide nanocomposites (ZnFe2O4) via the hydrothermal/solvothermal method typically involves the following key steps:

Precursor Preparation: The process begins with preparing a solution containing the metal precursors. Common precursors for zinc are zinc nitrate (Zn(NO3)2) or zinc acetate (Zn(CH3COO)2). For iron, iron nitrate (Fe(NO3)3) or iron chloride (FeCl3) are frequently used. These salts are dissolved in a solvent (e.g., deionized water for hydrothermal, or an organic solvent for solvothermal) along with a base (like sodium hydroxide or ammonia) to control the pH and facilitate precipitation.

Reaction: The precursor solution is transferred to a Teflon-lined stainless steel autoclave. The autoclave is then sealed and heated in an oven to a specific temperature, typically ranging from 120∘ C to 240∘ C, for a set period of time (e.g., a few hours to a day). The pressure inside the vessel increases due to the heating, creating the necessary conditions for the reaction. During this stage, the metal ions react and precipitate, leading to the formation of the nanocomposite particles.

Post-Synthesis Treatment: After the reaction is complete and the autoclave has cooled, the resulting product is collected. It's usually a solid precipitate that needs to be thoroughly washed with deionized water and/or ethanol to remove any unreacted precursors or byproducts. The final step is typically drying the powder in an oven. Sometimes, an additional calcination step at high temperatures (e.g., 400∘ C−700∘ C) is performed to improve crystallinity and remove any remaining organic impurities.

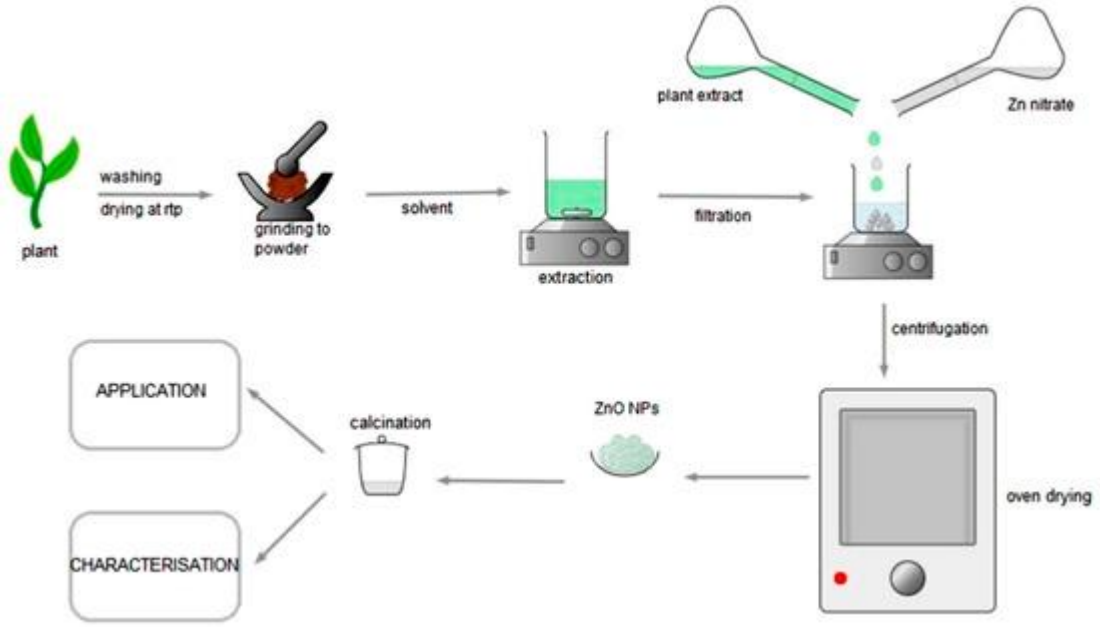

Figure 3. Schematic of ZnO-NPs synthesis procedure

This synthetic route offers several significant advantages for creating zinc iron oxide nanocomposites:

Controlled Nanostructure: The method allows for fine-tuning the size, morphology, and crystallinity of the nanoparticles. By adjusting parameters like temperature, pressure, reaction time, precursor concentration, and solvent, researchers can synthesize various shapes, including spheres, rods, or flakes. This is crucial as the properties of nanomaterials are highly dependent on their size and shape.

High Purity: The sealed environment of the autoclave minimizes the risk of contamination from the surrounding atmosphere. This leads to the synthesis of high-purity materials with fewer defects.

Energy Efficiency: Compared to traditional high-temperature solid-state reactions, the hydrothermal/solvothermal method typically requires lower reaction temperatures. This can lead to reduced energy consumption.

Single-Step Process: In many cases, the method can produce crystalline materials directly from the solution in a single step, without the need for post-synthesis annealing at extremely high temperatures.

The hydrothermal and solvothermal methods are powerful and versatile techniques for synthesizing zinc iron oxide nanocomposites. Their ability to precisely control the reaction conditions and the resulting nanostructure makes them a preferred choice for researchers in materials science and nanotechnology. By manipulating parameters, it's possible to create materials with tailored properties for specific applications, such as catalysis, gas sensing, or energy storage. The synthesis of high-quality zinc iron oxide nanocomposites is a testament to the efficacy of these methods in modern materials chemistry

The sol-gel method is a versatile and widely used technique for synthesizing zinc iron oxide ($ZnFe_2O_4$) nanocomposites. This process is favored for its ability to produce highly homogeneous materials with precise control over their composition, size, and morphology, which are crucial for their electronic, magnetic, and catalytic applications. The method involves a series of chemical reactions that transform a liquid precursor solution (sol) into a solid gel, which is then dried and heated to form the final material.

The synthesis of zinc iron oxide nanocomposites via the sol-gel method can be broken down into three main stages: sol formation, gelation, and thermal treatment. This initial stage involves preparing a homogeneous solution (the sol) of the precursor materials. Typically, metal salts like zinc acetate and iron nitrate are dissolved in a solvent, often deionized water or an alcohol. A complexing agent, such as citric acid or ethylene glycol, is usually added to chelate the metal ions. This is a critical step as it prevents the selective precipitation of one metal over the other, ensuring a uniform distribution of zinc and iron ions throughout the solution. The chelation process also helps to control the size and morphology of the nanoparticles in the final product.

Once the sol is formed, it is left to undergo a condensation and polymerization process. The chelated metal ions react to form a three-dimensional network. This process transforms the liquid sol into a rigid, transparent gel. The solvent molecules are trapped within the pores of this gel network. The time required for gelation can be influenced by factors such as the concentration of the precursors, the solvent, pH, and temperature.

The final stage involves a two-step thermal treatment. First, the gel is dried at a relatively low temperature to remove the trapped solvent. This drying process is often slow to prevent cracking and maintain the integrity of the gel network. The resulting porous solid is called a xerogel. Second, the xerogel is subjected to a high-temperature calcination process. This heating step burns off any remaining organic components (like the complexing agent) and promotes the crystallization of the amorphous metal oxide network into the desired crystalline zinc iron oxide (ZnFe2O4) nanocomposite. The calcination temperature and duration are crucial for controlling the final particle size, crystallinity, and phase purity of the material.

The sol-gel method offers significant advantages over other synthesis techniques, such as co-precipitation or hydrothermal methods. Its ability to create highly homogeneous mixtures at the atomic level ensures a uniform product. The low processing temperatures during the initial stages make it possible to incorporate heat-sensitive materials, and the ability to control particle size and morphology allows for the tuning of the nanocomposite's properties.

Zinc iron oxide nanocomposites synthesized via the sol-gel method have numerous applications. Their magnetic properties make them suitable for use in magnetic storage devices and biomedical applications, such as drug delivery and hyperthermia treatments. Their catalytic properties are exploited in environmental remediation and as photocatalysts for water splitting and the degradation of pollutants. Furthermore, their semiconducting nature makes them candidates for use in gas sensors and spintronic devices. The tunability of their properties through the sol-gel process allows for their optimization for specific applications, making this synthesis method highly valuable in materials science and engineering.

Combustion synthesis, also known as self-propagating high-temperature synthesis (SHS), is a powerful technique for creating advanced materials. It's a rapid and cost-effective method that uses an exothermic (heat-releasing) reaction between chemical precursors to form the desired product. The process is self-sustaining, meaning that once the reaction is initiated, it continues on its own without external heating. This method is particularly useful for creating nanocomposites because the quick, high-temperature reaction limits particle growth, resulting in very small, uniform nanocrystals.

The quality of the final product largely depends on the choice of precursors, which are the starting materials. For zinc iron oxide nanocomposites, the main precursors are:

Metal Salts: These are the sources of the metal ions. Common choices include zinc nitrate ($Zn(NO_3)_2 \cdot 6H_2O$) and iron nitrate ($Fe(NO_3)_3 \cdot 9H_2O$). They dissolve in a solvent (usually water) to form a homogeneous solution.

Fuel: The fuel is a crucial component that provides the energy for the exothermic reaction. It acts as both a reducing agent and a complexing agent. Popular fuels include urea ($CO(NH_2)_2$), glycine ($C_2H_5NO_2$), or citric acid ($C_6H_8O_7$). The fuel-to-oxidizer ratio (the ratio of fuel to the metal nitrates) is a critical parameter that controls the reaction temperature and the characteristics of the resulting nanocomposite.

The combustion synthesis procedure for zinc iron oxide nanocomposites generally involves three main steps:

Preparation of the Precursor Solution: The metal nitrates and the chosen fuel are dissolved in a solvent (typically deionized water) to create a clear, homogeneous solution. The solution is then heated and stirred to ensure complete dissolution and mixing of the components.

Ignition of the Reaction: As the water evaporates, the solution becomes more concentrated and viscous, eventually forming a gel. Further heating causes the mixture to ignite, leading to a vigorous, self-propagating combustion reaction. The fuel burns rapidly, releasing a large amount of heat and gases. This rapid reaction synthesizes the zinc iron oxide nanoparticles.

Formation of the Nanocomposite: The heat from the combustion reaction is intense enough to decompose the nitrates and the fuel, leading to the formation of the desired zinc iron oxide phases, such as zinc ferrite ($ZnFe_2O_4$) or other mixed zinc iron oxides. The quick nature of the reaction prevents the nanocrystals from growing too large. The final product is a loose, fine powder with a very high surface area.

The combustion synthesis method is a highly effective technique for producing zinc iron oxide nanocomposites. Its simplicity, speed, and ability to produce high-quality, nanoscale materials make it a preferred choice for many research and industrial applications. The key to success lies in the careful selection of precursors and the optimization of the fuel-to-oxidizer ratio to control the combustion reaction and achieve the desired material properties.

Findings and Discussion

X-ray Diffraction (XRD) is an essential non-destructive technique for characterizing the structural properties of zinc iron oxide nanocomposites. It provides critical information on the material's crystallinity, phase composition, crystallite size, and lattice parameters. The principles of XRD are based on Bragg's law, which relates the spacing between atomic planes in a crystal to the angles at which X-rays are diffracted.

XRD operates on the principle of Bragg's Law, given by the equation: $n\lambda=2d\sin\theta$ where n is an integer representing the order of diffraction, λ is the wavelength of the X-rays, d is the interplanar spacing of the crystal lattice, and θ is the angle of incidence of the X-ray beam.

When an X-ray beam hits a crystalline sample, it interacts with the electrons of the atoms, causing them to scatter. In a crystal, atoms are arranged in periodic planes. If the scattered waves from different planes interfere constructively, a diffraction peak is produced at a specific angle (2θ). By rotating the sample and detector, a diffraction pattern is recorded, which is a plot of diffraction intensity versus the scattering angle (2θ).

UV-Vis spectroscopy is a valuable tool for characterizing zinc iron oxide nanocomposites, providing insights into their optical properties and electronic structure. This technique measures the absorption or transmission of light by a sample as a function of wavelength in the ultraviolet (UV) and visible (Vis) regions of the electromagnetic spectrum. The interaction of photons with the electrons in the nanocomposite allows for the determination of key parameters like the bandgap, which is crucial for applications in photocatalysis, sensing, and optoelectronics.

The fundamental principle behind UV-Vis spectroscopy is the Beer-Lambert Law, which states that the absorbance of a solution is directly proportional to the concentration of the absorbing species and the path length of the light through the sample. In the case of solid-state materials like nanocomposites, the light absorption is related to the electronic transitions within the material. When a photon with sufficient energy strikes the nanocomposite, it can excite an electron from a lower energy level (valence band) to a higher energy level (conduction band). The energy required for this transition corresponds to the bandgap of the material.

For zinc iron oxide nanocomposites, UV-Vis spectroscopy is used to determine several important characteristics:

Bandgap Energy (Eg): The bandgap is a critical parameter that defines the energy required to excite an electron from the valence band to the conduction band. A lower bandgap means the material can absorb light at longer wavelengths (lower energy), making it more suitable for visible light applications. The bandgap is typically calculated from the absorption spectrum using the Tauc plot method. The Tauc plot relates the absorption coefficient (α) to the photon energy ($h\nu$) using the equation: $(\alpha h\nu)^n = A(h\nu - Eg)$, where A is a constant and n is an index that depends on the type of electronic transition ($n=2$ for direct transitions and $n=1/2$ for indirect transitions).

Surface Plasmon Resonance (SPR): In certain cases, especially with nanoscale metal components, the nanocomposite may exhibit a surface plasmon resonance peak in the visible region. This occurs due to the collective oscillation of conduction electrons in response to incident light. While zinc iron oxide itself doesn't have a strong SPR, the presence of other metallic nanoparticles in the composite can be detected this way.

Defects and Impurities: The presence of defects or impurities can introduce new energy levels within the bandgap, leading to additional absorption peaks or shoulders in the UV-Vis spectrum. These can be identified and analyzed to understand the structural integrity and quality of the nanocomposite.

Conclusion

The synthesis and characterization of zinc iron oxide nanocomposites represent a significant area of research in materials science. The ability to tailor their properties through different synthesis methods and to understand their structure through various characterization techniques opens up a plethora of opportunities for their application in various fields. Future research will likely focus on developing more efficient and environmentally friendly synthesis methods and exploring new applications for these fascinating materials.

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