

IMPROVED METAL NONPOROUS MATERIALS DERIVED FROM SURFACTANTS: SYNTHESIS, CHARACTERIZATION, AND APPLICATIONS

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ABSTRACT

When synthesizing metal nanoparticles, it is possible to regulate their size, shape, and dispersion stability by choosing the stabilizer with care and changing the molar ratio of stabilizer to precursor ions. Conversely, inadequate surface use may occur if the stabilizing chemicals used impede the nanoparticles' active regions. This study describes several methods for producing metal nanoparticles in a surfactant-free environment and provides examples of their use in catalysis and sensing. When discussing the manufacture of metal nanoparticles, the phrase "surfactant-free synthesis" does not imply that stabilizing agents such thiolate and phosphine compounds, surfactants, or polymers are not used. These metal nanoparticles are stabilized by the presence of solvents, simple ions from the reducing agents, or salts with a low molecular weight. Additionally, methods for creating metal nanoparticles without the use of surfactants are described, including laser ablation, photochemical, and ultrasonochemical synthesis. Due to the efficient utilization of their surfaces in SALDI-MS and other methods, metal nanoparticles prepared without surfactants, polymers, templates, or seeds are expected to exhibit excellent performance in sensing (surface-enhanced Raman scattering, SERS) and catalysis (electrocatalysis and synthetic catalysis).

Keywords: catalysis; nanoparticles; SALDI-MS; SERS; surfactant-free synthesis.

1. INTRODUCTION

The development of advanced materials has become a cornerstone of modern technology, enabling significant improvements across a variety of sectors such as energy storage, catalysis, environmental remediation, and electronics. Among these materials, nonporous metal materials have attracted considerable attention due to their unique surface properties, tunable structure, and wide range of industrial and scientific applications. Recent advancements in the synthesis of nonporous materials have leveraged the properties of surfactants to produce highly structured, efficient, and stable materials. This approach has proven to be a significant innovation in material science, providing a pathway for the development of materials with enhanced mechanical, chemical, and physical properties.

Importance and Role of Surfactants in Synthesis

Surfactants, compounds composed of both hydrophilic and hydrophobic parts, have long been utilized as essential agents in material synthesis. They are primarily known for their ability to reduce surface tension and form micelles in aqueous solutions. However, their utility goes far beyond surface-active properties. Surfactants have been identified as key components in the template-directed synthesis of nonporous metal materials, where they serve to guide the arrangement of metal ions into highly ordered structures. The amphiphilic nature of surfactants allows them to self-assemble into a variety of nanostructures such as micelles, vesicles, or liquid crystalline phases. These structures act as templates around which metal ions can deposit and crystallize into specific forms. The removal of the surfactant afterward leaves behind a well-ordered nonporous structure with enhanced surface characteristics. This templating effect offers precise control over the size, morphology, and porosity of the metal material, which is critical for applications that require specific surface properties such as catalysis and energy storage. One of the significant advantages of using surfactants in material synthesis is their ability to produce uniform and highly stable metal nonporous materials. The interaction between the surfactant molecules and metal ions facilitates the formation of highly interconnected frameworks with minimal defects. Moreover, by varying the surfactant type, concentration, and synthesis conditions, it is possible to fine-tune the characteristics of the resultant materials, such as their surface area, pore size, and overall stability. This versatility makes surfactant-derived nonporous materials particularly attractive for a wide range of applications where material performance is highly dependent on structural precision.

Synthesis of Metal Nonporous Materials

The synthesis of metal nonporous materials typically involves three critical steps: the formation of a surfactant template, the incorporation of metal precursors, and the removal of the surfactant to reveal the final nonporous structure. The first step is the self-assembly of the surfactant molecules into well-defined structures. This can be achieved by carefully selecting the type of surfactant and adjusting parameters such as temperature, pH, and ionic strength to promote the formation of specific micellar or vesicular structures. In the next step, metal precursors, which

are often metal salts or organometallic compounds, are introduced into the system. These metal ions interact with the surfactant templates, leading to their deposition along the surfactant surfaces. This interaction is guided by electrostatic forces, hydrophobic interactions, or specific binding affinities between the metal ions and surfactant molecules. As a result, the metal ions organize into highly structured nonporous frameworks that mirror the shape and arrangement of the surfactant template.

The final step in the synthesis process involves the removal of the surfactant, typically through calcination, solvent extraction, or other chemical treatments. Once the surfactant is removed, what remains is a nonporous metal material with a highly ordered structure and a large surface area. This material often exhibits enhanced mechanical stability and catalytic activity compared to similar materials synthesized without the use of surfactants. The precise control over the synthesis process allows for the production of materials with customizable properties, making them ideal for a wide array of high-performance applications.

Characterization Techniques

To fully understand the properties and potential applications of these metal nonporous materials, a comprehensive set of characterization techniques is employed. These techniques provide insight into the structural, morphological, and chemical properties of the synthesized materials. One of the primary methods used for structural analysis is X-ray diffraction (XRD), which reveals the crystalline nature of the material. XRD patterns can provide valuable information about the material's phase composition, crystallite size, and degree of crystallinity. The presence of well-defined peaks in the XRD spectrum is indicative of the ordered arrangement of metal ions, confirming the successful templating effect of the surfactant. Scanning electron microscopy (SEM) and transmission electron microscopy (TEM) are commonly used to examine the morphology and microstructure of the materials. SEM allows for the visualization of surface features, while TEM provides detailed images at the atomic level, enabling researchers to assess the arrangement of metal particles and the overall framework of the material. These techniques also help identify any defects or irregularities in the structure, which can affect the material's performance in various applications. Additionally, nitrogen adsorption-desorption isotherms are employed to measure the specific surface area, pore size distribution, and porosity of the material. This technique is critical for determining the material's suitability for applications such as catalysis and adsorption, where high surface area and well-defined pore structures are crucial. The Brunauer-Emmett-Teller (BET) method is often used to calculate surface area, while the Barrett-Joyner-Halenda (BJH) method helps estimate pore size distribution. Finally, Fourier-transform infrared spectroscopy (FTIR) and thermogravimetric analysis (TGA) are employed to analyze the chemical composition and thermal stability of the material. FTIR provides information on the functional groups present, confirming the successful removal of the surfactant, while TGA helps assess the thermal stability and decomposition behavior of the material, which is particularly important for applications in high-temperature environments.

Applications of Metal Nonporous Materials

Metal nonporous materials derived from surfactants have found applications in a wide range of fields due to their unique properties, such as high surface area, enhanced stability, and tunable porosity. These properties make them highly effective for catalysis, where the large surface area allows for increased interaction between the catalyst and reactants, leading to higher reaction rates. Metal nonporous catalysts are used in processes such as hydrogenation, oxidation, and pollutant degradation, playing a crucial role in both industrial and environmental applications. In the field of energy storage, these materials are utilized in supercapacitors and batteries due to their ability to store and release electrical energy efficiently. Their high surface area and structural stability allow for the rapid movement of ions, making them ideal for use in high-performance energy storage devices.

Furthermore, metal nonporous materials are gaining attention for their adsorption capabilities, particularly in environmental remediation. These materials can adsorb a wide range of pollutants, including heavy metals, organic compounds, and gases, providing a sustainable and efficient method for water and air purification. Their tunable porosity allows for the selective adsorption of specific contaminants, making them a versatile solution for addressing environmental challenges.

Classification of Nanoparticles

Organic Nanoparticles

One of the main components of organic nanoparticles (ONPs) is an organism with a molecular size of 100 nm or less [14]. This class includes well-known organic nanoparticles and polymers such as ferritin, micelles, dendrimers, and liposomes. Nanoparticles with a hollow center, or nanocapsule, are sensitive to heat and electromagnetic radiation (light) and are biodegradable and non-toxic [15]. Because of their distinct qualities, they are superior choices for medication administration. Their drug-carrying capacity, stability, and delivery systems—such as an entrapped drug or adsorbed drug system—influence their efficiency and area of application, regardless of the importance of size,

composition, surface shape, etc. [16]. Because of their effectiveness and the possibility of targeted medication administration, which involves injecting them into specific areas of the body, organic nanoparticles find several applications in biomedicine, including drug delivery systems [17].

Metal Oxide-Based Nanoparticles

Over the last few decades, metal oxides have become more popular among scientists. By mixing positive metallic ions with negative oxygen ions, ionic compounds known as metal oxides may be formed. Ionic connections are strong and last for a long time because of the electrostatic interactions between oxygen ions and positively charged metal ions. One example is the simple and rapid reaction between iron nanoparticles (Fe) and oxygen at room temperature; this reaction is much more reactive than that between iron oxide nanoparticles (Fe_2O_3). The properties of metal-based counterparts might be changed by oxide nanoparticles. Nanoparticles made of metal oxides are often used in applications that need more efficiency and reactivity. Al_2O_3 , silicon dioxide, zinc oxide, titanium dioxide, and zinc oxide are among the most frequently produced oxides. These nanoparticles are quite different from their metal equivalents in terms of their characteristics.

Carbon-Based Nanoparticles

Carbon-based nanoparticles (CBNPs) are a class of nanomaterials that have gained widespread attention in recent years due to their unique physical, chemical, and electronic properties. These nanoparticles, typically ranging in size from 1 to 100 nanometers, are composed of carbon atoms arranged in various forms, including spherical fullerenes, cylindrical carbon nanotubes (CNTs), graphene sheets, and carbon quantum dots (CQDs). Their exceptional versatility, combined with their tunable properties, has made them highly valuable in a broad range of applications, including electronics, catalysis, energy storage, drug delivery, and environmental remediation. The underlying theory behind carbon-based nanoparticles centers on their molecular structure, quantum confinement effects, and surface-to-volume ratio. These characteristics impart unique optical, electronic, mechanical, and thermal properties that differ significantly from their bulk counterparts. By understanding the fundamental properties and behavior of carbon atoms at the nanoscale, researchers can manipulate the synthesis and design of carbon-based nanoparticles for specific applications.

Molecular Structure and Allotropes of Carbon

Carbon is an element with remarkable versatility, owing to its ability to form four covalent bonds with other atoms. This enables carbon atoms to bond with themselves and other elements in a variety of configurations, resulting in different allotropes. The most well-known carbon allotropes at the nanoscale are fullerenes, carbon nanotubes, graphene, and carbon quantum dots. Fullerenes: Fullerenes are hollow, cage-like structures made entirely of carbon atoms. The most common fullerene, C_{60} , consists of 60 carbon atoms arranged in a spherical shape resembling a soccer ball. These closed structures exhibit high symmetry and are known for their ability to encapsulate other molecules, making them ideal candidates for drug delivery systems and molecular electronics. The discovery of fullerenes in 1985 marked the beginning of nanocarbon science, highlighting the ability of carbon atoms to form unique, stable structures at the nanoscale.

Carbon Nanotubes (CNTs): Carbon nanotubes are cylindrical nanostructures made of rolled-up graphene sheets. They can be single-walled (SWCNT) or multi-walled (MWCNT), depending on the number of graphene layers forming the tube. CNTs possess remarkable mechanical strength, thermal conductivity, and electrical properties, making them highly attractive for use in nanoelectronics, sensors, and materials science. The theoretical study of CNTs involves understanding the electronic structure of the rolled graphene sheet, which determines whether the nanotube behaves as a metallic or semiconducting material based on its chirality and diameter.

Graphene: Graphene is a single layer of carbon atoms arranged in a two-dimensional honeycomb lattice. It exhibits extraordinary electrical conductivity, flexibility, and mechanical strength. The theoretical foundation of graphene involves the study of its unique electronic band structure, particularly the Dirac points at which the conduction and valence bands meet. These points give rise to massless charge carriers that travel at high speeds, leading to graphene's superior conductivity. Graphene's large surface area and tunable properties make it suitable for a range of applications, from flexible electronics to energy storage devices.

Carbon Quantum Dots (CQDs): Carbon quantum dots are small, quasi-spherical nanoparticles with a diameter typically less than 10 nanometers. They exhibit size-dependent optical properties due to quantum confinement effects, which arise when the particle size approaches the exciton Bohr radius. Quantum confinement leads to the discretization of energy levels and shifts in the optical absorption and emission spectra, making CQDs highly fluorescent and useful in bioimaging, sensing, and light-emitting applications. Fullerenes One of the most famous and extensively used fullerenes, Buckminster fullerene is C_{60} . With 60 carbon atoms arranged in a cage-like fashion, each with three bonds, it resembles a soccer ball in form [36]. Twenty hexagons and twelve pentagons make up the C_{60}

structure. Two well-established features of this structure are resonance stabilization and icosahedral symmetry. The area of material science finds use for it because of its unique physicochemical properties. In recent years, several areas of nanoscience and nanotechnology have made extensive use of nanorods, nanotubes, and nanosheets, all of which are based on C60. C60 has several various applications and can accelerate the reactions of many different chemicals. Its unique characteristics make it well-suited for incorporation into systems with the aim of enhancing targeted behaviors. C60 is amenable to covalent, endohedral, and supramolecular transformations, allowing for molecular manipulation and the creation of polymeric materials with potential environmental applications.

Quantum Confinement Effects

One of the defining characteristics of carbon-based nanoparticles is the quantum confinement effect, which refers to the spatial restriction of electrons within a particle when its size is comparable to the de Broglie wavelength of the electron. In bulk materials, electrons can move freely within the crystal lattice, leading to a continuous range of energy states. However, when the size of the material is reduced to the nanoscale, the movement of electrons becomes confined to discrete energy levels.

The quantum confinement effect plays a significant role in determining the optical and electronic properties of carbon-based nanoparticles, particularly in materials like carbon quantum dots and small graphene fragments. As the size of the nanoparticle decreases, the energy gap between discrete energy levels increases, leading to shifts in the material's absorption and emission spectra. This effect enables precise control over the optical properties of CBNPs, making them ideal for applications in optoelectronics and photonics.

In the case of carbon quantum dots, the quantum confinement effect is responsible for their tunable fluorescence. By adjusting the size of the CQDs, researchers can control the wavelength of light emitted when the dots are excited by an external light source. This property is highly useful for applications such as bioimaging and light-emitting devices, where specific wavelengths of light are needed for accurate visualization or signal transmission.

Surface-to-Volume Ratio

Nanoparticles, by their very nature, have an extremely high surface-to-volume ratio compared to bulk materials. This increased surface area is particularly important in carbon-based nanoparticles, where surface interactions play a critical role in determining the material's overall behavior. The large surface area of CBNPs enhances their interaction with other molecules, making them ideal candidates for catalysis, adsorption, and drug delivery.

For example, in catalysis, the high surface area of carbon nanotubes or graphene sheets allows for more active sites where catalytic reactions can occur, leading to enhanced catalytic efficiency. Similarly, in environmental applications, CBNPs can be used as adsorbents to capture pollutants due to their high surface area and ability to interact with various chemical species.

The surface chemistry of carbon-based nanoparticles is also important for their functionalization and integration into composite materials. By modifying the surface of CBNPs with specific functional groups or molecules, researchers can tailor their properties for targeted applications. For instance, the surface functionalization of carbon nanotubes with oxygen-containing groups can improve their dispersibility in water and enhance their interaction with biological systems for drug delivery or biosensing applications.

Carbon Nanotubes (CNTs)

Carbon nanotubes, or CNTs, are a very flexible carbon allotrope. It has a designed and manufactured structure of long, cylindrical, and tubular graphene sheets. The quantity of concentrically interlocked nanotubes required to construct multi-walled carbon nanotubes differentiates them from single-walled carbon nanotubes. Unlike SWCNTs, which have a maximum diameter of 3 nm, MWCNTs may reach sizes of over 100 nm. Mechanically stronger than SWCNTs, MWCNTs are composed of many layers of carbon atoms. Compared to common metals such as iron and steel, CNTs have much better tensile strength and Young's modulus. Sensor applications benefit greatly from SWCNTs due to their remarkable twisting capabilities. Due to their extraordinary durability, MWCNTs are used in composites research. An assortment of mechanical, electrical, chemical, and biological uses for CNTs have been documented. Numerous electromagnetic domains rely on carbon nanotubes, such as electrochemistry, energy storage, vacuum microelectronics, and electron field emission systems. Because of their flawless roundness and pinpoint accuracy, they dominate the electrical industry. Moreover, hydrogen might be stored via carbon nanotubes. They are ideal for this purpose because to their high absorption rate. As a reinforcing and filler material, carbon nanotubes (CNTs) are often used to reduce the weight of mechanical composites.

Activated Carbon or Charcoal

Carbon that has been processed to create very small pores and a very low volume is known as charcoal or activated carbon. The goal of synthesizing any given substance is to increase its surface area so that it may be more effectively

used in chemical processes or adsorbed. Therefore, activated carbon is a common adsorbent in water purification techniques for eliminating impurities. It is often used for treating water, extracting minerals from water, and purifying gases and colors. The problem is that the process of removal is inefficient. A functional type of activated carbon called nanoporous activated carbon may be used to get the required efficiency. Nanoporous structures are most often made of carbon. Hydrogen and oxygen are also present, albeit in far smaller proportions. Possible inorganic components, sulphur, nitrogen, and phosphorus content depends on precursor, manufacturing method, and post-synthesis processing. As one examines the nanoporous surface, oxygen groups become more apparent. Nanoporous activated carbon is very beneficial due to its pores (micropores or (micro + mesopores)) and heteroatoms (which include sulfur, nitrogen, and oxygen).

Sol-Gel Method

As a soft chemical process, the sol-gel technique is exceedingly versatile and is often used to manufacture metal oxides, ceramics, and glasses. A wide variety of ceramics and glasses, including thin film coatings, microporous inorganic membranes, ultrafine or spherical powders, and ceramic fibers, may be commercially available.

The sol-gel technique often begins with metal alkoxides or organometallic inorganic salts. The precursor may be converted into a sol or colloidal suspension by a sequence of processes that includes hydrolysis and polycondensation. Molecules in a system may go from being a homogeneous liquid (the "sol") to a solid (the "gel") via the sol-gel process, which occurs at room temperature and pressure. Producing the metal oxide nanopowder requires drying the gel and subjecting it to calcination at varying temperatures. It is possible to alter the shape, morphology, and textural characteristics of the manufactured materials using the sol-gel process.

The sol-gel technique may manufacture metastable materials, has the ability to lower working temperatures, and improves product purity and compositional homogeneity compared to high-temperature alternatives. This process also affects the particle shape when the molecular precursor goes through chemical transformation into the final oxidic network. The sol-gel approach has been used by several research groups to produce nanoscale alumina and iron oxide.

With this method, you can process a wide range of materials. It has many advantages, such as creating metal and ceramic nanomaterial at temperatures between 70 and 320 degrees Celsius, getting atomically homogeneous final products, controlling composition at the molecular level, and getting porous, high-surface-area materials. In addition to producing more pure end products, utilizing high-purity reagents allows for the synthesis of materials with complicated components. Using physical deposition techniques to achieve thicknesses more than a micron is rather challenging; however, this problem is solved by the sol-gel approach. It is feasible to wrap intricate geometries using synthetic materials with complex compositions. Hydrolysis and polycondensation are typical methods for producing sols from inorganic metal salts or metal-organic complexes. Many other types of ceramic materials may be made from the sol by further processing. Thin films may be created using either spin coating or dip coating. When you pour Sol into a mold, it will turn into a gel-like substance. Due to volumetric shrinkage, a solid and inflexible structure is achieved once the gel has dried. To achieve nanoscale porosity, one must carefully regulate the drying conditions of the gel. As an advantage over normal porosity, nanoporosity has a larger specific surface area. As an example, the porosity of a nanoporous carbon material has the potential to absorb and retain hydrogen.

The main problems with the sol-gel method based on metal alkoxides are their sensitivity to water and the lack of commercially available precursors that are suitable for mixed-metal oxide synthesis. It may be difficult to sol-gel synthesize mixed oxides from combinations of alkoxides because the constituent parts have different hydrolysis susceptibilities. Moreover, the advantages of improved homogeneity may be diminished due to component dispersion and mixed phases in the final product caused by the hydrolysis of the alkoxides.

2. CONCLUSION

The synthesis of metal nonporous materials using surfactants as templates represents a significant advancement in material science. By leveraging the self-assembly properties of surfactants, researchers can design materials with highly controlled structures, resulting in enhanced performance across a wide range of applications. From catalysis and energy storage to environmental remediation, these materials hold immense potential for driving technological innovation and sustainability in the coming decades. With continued research and development, the scope of their applications is expected to expand, paving the way for new discoveries and practical implementations across various industries.

3. REFERENCES

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