Synthesis and Characterization of Surfactant-Based Metal Nonporous Materials and their Enhanced Applications

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Abstract: Metal nanoparticles may have their size, shape, and dispersion stability controlled by carefully selecting the stabilizer and adjusting the molar ratio of stabilizer to precursor ions during synthesis. On the other hand, the stabilizing chemicals utilized could obstruct the active areas on the surfaces of the nanoparticles, leading to inefficient surface use. Several techniques for generating metal nanoparticles in a surfactant-free solution are detailed in this paper, along with examples of their use in sensing and catalysis. The term "surfactant-free synthesis" does not mean that no stabilizing agents, such as thiolate and phosphine compounds, surfactants, or polymers, are used in the preparation of the metal nanoparticles. The solvents, simple ions of the reducing agents, or low-molecular-weight salts used stabilize these metal nanoparticles. Laser ablation, photochemical, and ultrasonochemical synthesis techniques for the surfactant-free creation of metal nanoparticles are also detailed. It is anticipated that metal nanoparticles prepared without surfactants, polymers, templates, or seeds will demonstrate excellent performance in sensing (surface-enhanced Raman scattering, SERS), and catalysis (electrocatalysis and synthetic catalysis) due to the efficient utilization of their surfaces in SALDI-MS and other methods.

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

Introduction

The size of metal nanoparticles typically falls between 1 and 100 nm. Metal nanoparticles have exceptional physiochemical characteristics in the areas of optics, magnetism, and chemical reactions (catalysis) due to their large surface area, high surface energy, and quantum confinement. In comparison to their bulk equivalents, these nanoparticles exhibit reduced electric/thermal conductivities and melting temperatures. As the size of metal nanoparticles shrinks and the number of atoms present at their surfaces increases, their physicochemical characteristics undergo changes. Not only do these size-based effects matter, but so do the nanoparticles' morphology, composition (i.e., whether they are metal or an alloy), and agglomeration, all of which influence the nanoparticles' physical and chemical properties. Research on metal nanoparticles is becoming more popular among scientists as a result of the emergence of controlled solution-based methods for producing these particles. Biomedical[1], optical[2,3], magnetic[4], catalytic[5-7], sensing[8-10], energy[11], and electrical applications[12–14] are only a few of the many possible uses for these nanoparticles. Furthermore, hybrids incorporating metal nanoparticles with synthetic polymers, biomolecules, and semiconductors have also been created [15–17].

A new category of metal nanoclusters (NCs), measuring less than 2 nm in size, has emerged as a result of recent developments in the solution-based production of metal nanoparticles, including those of Ag or Au. The fact that these metal NCs bridge the gap between the behavior of metals at the atomic and nanoparticle levels makes them quite intriguing. Metal NCs with sizes close to the Fermi wavelength exhibit molecule-like characteristics such as size-dependent fluorescence, distinct electronic states, and specialized catalytic activity.

Creation, organization, and characteristics of atomically accurate Au NCs possessing rigorous stoichiometry Chemical reduction of precursor ions in solution by a reducing agent is one solution-based approach for synthesizing metal nanoparticles that has been developed in the last 20 years. This allows for control over the size and form of the nanoparticles. In order to create metal nanoparticles in a solution, three common ingredients are a reducing agent, a stabilizing agent, and a soluble metal salt. Metal nanoparticles are often produced using reducing agents such sodium borohydride, citrate ions, and alcohols. To stop the nanoparticle from growing or clumping together, stabilizing chemicals including thiol, phosphine, amine compounds, surfactants, and polymers attach to its surface. Beyond just keeping the metal nanoparticles in place, the stabilizing agent can also impart additional functions, such as surface reversal between hydrophilic and hydrophobic particles, biomaterial coupling for recognition, delivery, and manipulation in biology, and the production of building blocks for devices. The two most popular approaches to manufacturing metal nanoparticles using a solution are these two procedures. The "Turkevich method" is the first, and it's a water-based process that uses citrate ions to reduce gold ions in water . The second approach, known as the "Brust method," is based on organic solutions and comprises reducing gold ions in organic media with $NaBH_4$ while a phase-transfer agent is present . Metal nanoparticle size and form are controlled by the stabilizer used in synthesis and the molar ratio of stabilizer to precursor ions. On the other hand, the stabilizing substance could at times obstruct the nanoparticles' active locations, leading to inefficient surface utilisation. For instance, catalytic activity could be reduced if the active sites are blocked. The production of metallic nanoparticles using a solution-based method that does not use surfactants is detailed in this review. Additionally, we detail the many uses for these nanoparticles. The phrase "surfactant free" does not suggest the production of nanoparticles made of pure metal. The preparation of metal nanoparticles in surfactant-free solution-based synthesis does not include the use of any extra stabilizing agents, including thiolate and phosphine compounds, surfactants, or polymers. Solvents or ions of reducing agents or salts stabilize these metal nanoparticles. This review does not address the synthesis of nanoparticles in the vapor phase, even though procedures involving the vapor phase are necessary to create really bare metal nanoparticles. Various surfactant-free synthesis processes are reviewed in the first half of this paper, and their applications are described in the second. Section one details the several ways in which solvent-stabilized metal nanoparticles may be produced, with the solvents serving as reaction medium, reducing agent, and stabilizer all in one. Nanoparticles of metal that have been stabilized by ions are thereafter detailed. The simple ions have three functions: reducing, protecting, and shaping. After that, methods for synthesising metal nanoparticles without the need of surfactants are described, using photochemical, ultrasonochemical, and laser ablation-mediated approaches. At last, the metal nanoparticles that were produced in this way are detailed along with their uses in sensing (surface-enhanced Raman scattering, SALDI-MS), and catalysis (electrocatalysis and synthetic catalysis). Due to the efficient exploitation of their surfaces, metal nanoparticles synthesized without surfactants, polymers, templates, or seeds demonstrated excellent performance in catalysis and sensing.

Carbon compounds with big pores, a high surface area, chemical inertness, and electrical conducting properties1 have piqued a lot of people's curiosity. From separation and adsorption to catalytic processes and catalyst supports, these nanostructure materials have shown to be invaluable in several domains[2]-[6.] A lot of the progress in carbon technology has been about refining and expanding upon current manufacturing processes and synthetic procedures.. Amphiphilic surfactants or block copolymers are used as soft templates in a multi-step synthetic technique that has recently produced several forms of organized nanoporous carbons. One way to create materials with complex multi-component structures is by using amphiphilic surfactants. Another way is to use block copolymer self-assembly. These methods allow for the formation of organic and inorganic frameworks, respectively, and are held together by weak non-covalent interactions.

A number of synthesis techniques are being worked on or developed with the goal of improving properties and reducing production costs [8,9]. To enhance the chemical, optical, mechanical, and physical characteristics of nanoparticles, process-specific nanoparticles are synthesized by combining several methods [10]. Technological progress has allowed scientists to better understand nanoparticles and identify potential uses for them [11]. Nowadays, nanoparticles are ubiquitous, showing up in everything from electronics to renewable energy sources to aircraft. Nanotechnology holds the key to a sustainable and prosperous future.

Extensive research has led to the efficient synthesis of several nanoparticle kinds, including composite nanoparticles, metal oxides, and perovskites. One new class of nanomaterials with promising photocatalytic capabilities is the heterojunction structure and hybrid semiconductor nanoparticles. Deposition of Au metal onto colloidal CdSe nanorods was the first demonstration of the hybrid system. The use of sophisticated synthetic methods for manipulating the shape, size, location, and chemical make-up of different parts. Hydrogen production via clean solar-to-fuel conversion has been achieved by these HNPs by photocatalytic water splitting. When it comes to photocatalytic CO2 reduction, it's really crucial. Many environmental processes, including water purification, trash treatment, and antibiotic activities, rely on HNPs' photocatalytic capability [12].

The ecology is suffering greatly due to the overconsumption of medications and personal care products. Photocatalysts with novel heterojunction topologies, including Cd0.5Zn0.5 nanorods/BiOCl microspheres, are being developed. These materials have a high redox power, a cheap cost, and a high rate of electron-hole segregation. Cd0.5Zn0.5S has a wealth of resources, a wide range of solar light responses, high reduction power, and outstanding photocatalytic potential in this structure. Carrier reunion, low oxidative activity, and photo corrosion, however, reduce its photocatalytic effectiveness. Eco-friendliness, easy defect engineering, robust physical stability, and distinctive photo/electric characteristics are some of the photocatalytic activities shown by BiOCl. Nevertheless, recombination of photogenerated electron/hole pairs and a high band gap (3.2-3.4) are also issues. Heterojunction structures are formed to address these issues. According to research, the photoactivity of a Cd0.5Zn0.5S/BiOCl heterojunction is 9.6 times higher than that of BiOCl alone, and 2.8 times higher than that of individual Cd0.5Zn0.5S [13].

Classification of Nanoparticles

Organic Nanoparticles

Organisms with a molecular size of 100 nm or less are the building blocks of organic nanoparticles (ONPs) [14]. Ferritin, micelles, dendrimers, and liposomes are all examples of famous organic nanoparticles or polymers that fall under this category. In addition to being biodegradable and non-toxic, nanoparticles having a hollow core called a nanocapsule, such as micelles and liposomes, are heat and electromagnetic radiation (light) sensitive [15]. They are better options for medicine delivery due to their unique characteristics. Despite the significance of size, composition, surface form, etc., their efficiency and area of application are affected by their drug-carrying capacity, stability, and delivery systems, such as an entrapped drug or adsorbed drug system [16]. Organic nanoparticles have many uses in biomedicine, such as in drug delivery systems, due to their efficiency and the fact that they may be injected into particular parts of the body (a technique called targeted drug delivery) [17].

Metal Oxide-Based Nanoparticles

Metal oxides have garnered increasing attention from researchers in recent decades. It is possible to create ionic compounds called metal oxides by combining positive metallic ions with negative oxygen ions. Thanks to electrostatic interactions between oxygen ions and positively charged metal ions, ionic connections are robust and long-lasting . As an example, iron nanoparticles (Fe) are much more reactive than iron oxide nanoparticles (Fe_2O_3) when exposed to oxygen at ambient temperature, a process that is quite simple and quick. Nanoparticles made of oxides may alter the characteristics of their metal-based analogues. It is common practice to produce metal oxide nanoparticles for use in applications requiring increased reactivity and efficiency . Some of the most common oxides synthesized are silicon dioxide (SiO2), titanium dioxide (TiO_2), zinc oxide (ZnO), and aluminum oxide (Al_2O_3). These nanoparticles exhibit exceptional properties in contrast to their metal counterparts.

Carbon-Based Nanoparticles

Carbon has been essential to the growth of human civilisation on our planet. It produces bonds of unparalleled strength when mixed with other materials. A vast array of carbon-based nanomaterials have been developed during the last few decades, thanks to various synthesis techniques. They have found use in a wide variety of fields due to their peculiar form and unique set of characteristics. Energy storage and production, water and wastewater treatment, and biological applications are just a few of the many possible uses for carbon-based nanomaterials. Carbon may assume a wide variety of allotropic forms. Graphite, diamond, and buckminsterfullerene are all minerals that may exist in more than one form. When it comes to thermodynamic stability, graphite is head and shoulders above the others. Its great conductivity makes it useful in many electrical contexts, such as solar panels, batteries, electrodes, and more. Layers of graphene are what give graphite its distinctive appearance. Graphene, a novel form of carbon, is a two-dimensional sheet with an atomic honeycomb structure structured in a single layer. Because of its great strength, it is a valuable component in the production of various carbon nanoparticles. Another new kind of carbon is carbon nanotubes, or CNTs. Graphene, CNTs, and fullerenes are all synthesised in various methods, yet they share chemical and physical features. Given their age, fullerene-based derivatives and composites seem like the furthest-fetched idea to develop. Regardless, carbon nanotubes (CNTs) and graphene hold great promise as alternatives in many different areas, and there is a great deal of room for further research into these materials. Adsorption, separation, catalytic reactions, and countless more fields have shown activated carbon's superiority as an adsorbent.

Fullerenes

One of the most famous and extensively used fullerenes, Buckminster fullerene is C60. 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 C60 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.

Graphene and Graphene Oxide (GO)

Nanocomposites made of polymers have discovered graphene to be an advantageous component of these materials. Incredibly high-quality mechanical, electrical, and molecular barriers are to blame. Problems with agglomeration, restricted solubility, and the complicated bottom-up production are only a few of the difficulties with pure graphene. By following this simple top-down method, carbon sources may be converted into graphene oxide and other chemically related molecules. These substitutes for graphene are appropriate since they can be easily produced. The diffusion of functionalized oxygen groups onto their structure allows for both their considerable solubility and rapid surface modification. Also, nanocomposite polymers work well with GO as a filler. This is due to its exceptional characteristics and its ability to distribute well in polymer matrices. In order to prevent gas molecules from passing through, sp2 carbon atoms arrange themselves in a compact configuration. Therefore, it is widely used as a corrosion-resistant material, a packaging material, and a shield for sensitive electronics . The creation of stimuli-responsive materials makes advantage of GO because of its distinctive hydrophilic, electrical, and thermal characteristics.

Carbon Nanotubes (CNTs)

One very versatile allotrope of carbon is carbon nanotubes, or CNTs. It features a long, cylindrical, and tubular construction made of graphene sheets that were planned and produced. The difference between multi-walled and single-walled carbon nanotubes lies in the number of concentrically interlocked nanotubes used to make them. While MWCNTs may reach diameters more than 100 nm, SWCNTs are limited to a maximum of 3 nm. Due to the existence of several carbon atom layers, MWCNTs possess a higher mechanical strength compared to SWCNTs. The tensile strength and Young's modulus of CNTs are far higher than those of typical metals like steel and iron. Because of their exceptional twisting characteristics, SWCNTs are ideal for use as sensors. Composites research makes use of MWCNTs because of their exceptional endurance. There have been reports of CNTs being used in mechanical, electrical, chemical, and biological applications . Carbon nanotubes are vital in many electromagnetic fields, including vacuum microelectronics, electron field emission systems, energy storage, and electrochemistry. They rule the electrical sector with their perfect roundness and precise precision. Carbon nanotubes may also store hydrogen. Their high absorption rate makes them perfect for this use. To make mechanical composites lighter, carbon nanotubes (CNTs) are often used as reinforcing and filler materials.

Activated Carbon or Charcoal

Charcoal, or activated carbon, is a kind of carbon that has undergone processing to produce very tiny pores and a very low volume. Making something with a large surface area for use in chemical reactions or adsorption is why it is synthesized. As a result, activated carbon is often used as an adsorbent in processes that purify water by removing contaminants. Its typical applications include color and gas purification, mineral water extraction, and water treatment. The issue is that the removal is not very efficient. To achieve the necessary efficiency, one may employ nanoporous activated carbon, a functional form of activated carbon. Carbon is the most used material for nanoporous structures. In lesser amounts, you may also find hydrogen and oxygen. Based on the precursor, manufacturing technique, and post-synthesis processing, inorganic components, nitrogen, sulfur, and phosphorus might potentially be present. Oxygen groups stand out the most when one looks at the nanoporous surface. The excellent pore structure (micropores or (micro + mesopores)) and heteroatoms (containing sulfur, nitrogen, and oxygen) of nanoporous activated carbon make it very useful.

Sol–Gel Method

Metal oxides, ceramics, and glasses are all often synthesized using the sol-gel method, a soft chemical process that is very versatile. Ceramic fibers, thin film coatings, microporous inorganic membranes, ultrafine or spherical powders, and many more types of ceramics and glasses may be commercially accessible.

Metal alkoxides and organometallic inorganic salts are common starting materials for sol-gel processes. A sol or colloidal suspension may be produced from the precursor by a series of reactions including hydrolysis and polycondensation. The sol-gel process, which takes place at ambient temperature and pressure, transforms a system's molecules from a uniform liquid (or "sol") into a solid (or "gel"). Once the gel is prepared, it is dried and calcined at different temperatures to produce the metal oxide nanopowder. With the sol-gel method, one may modify the form, morphology, and textural properties of the materials that are produced.

In comparison to high-temperature approaches, the sol-gel process has several advantages, such as reduced working temperatures, improved product purity and compositional uniformity, and the capacity to produce metastable materials. As the molecular precursor undergoes chemical transformation into the final oxidic network, this process also influences the particle shape. For the production of nanoscale alumina and iron oxide, many research groups have previously reported using the sol-gel method.

A wide range of materials can be processed using this method, which has many advantages, including the ability to create metal and ceramic nanomaterial at temperatures ranging from 70 to 320 degrees Celsius, achieving final product homogeneity on an atomic level, controlling composition on a molecular scale, and obtaining high-surface-area materials with porosity. The synthesis of materials with complex components is also made possible by using high-purity reagents, which ultimately results in more pure final products. Achieving thicknesses more than a micron using physical deposition methods is rather tough; however, the sol-gel approach overcomes this challenge. It is possible to synthesize materials with complicated compositions and apply coatings over complex geometries. It is common practice to create sols from inorganic metal salts or metal-organic complexes by using hydrolysis and polycondensation processes. Further processing of the sol allows for the formation of a wide range of ceramic materials. Two ways to make thin films are spin coating and dip coating. Filling a mold with Sol causes it to solidify into a wet gel.After drying the gel, a solid and rigid structure is obtained as a result of volumetric shrinkage. It should be noted that by meticulously controlling the gel's drying conditions, a porosity on the nanoscale may be accomplished. The increased specific surface area compared to conventional porosity is one advantage of nanoporosity. For example, a nanoporous carbon material's porosity may capture and store hydrogen.

Water sensitivity and an absence of suitable commercially accessible precursors, particularly for mixed-metal oxides, are the primary issues in using the sol-gel process based on metal alkoxides. Mixed oxides from alkoxide combinations may be difficult to synthesize sol-gelly due to the varying hydrolysis susceptibilities of the constituent parts. Furthermore, the hydrolysis of the alkoxides may cause component dispersion and mixed phases in the end product, therefore reducing the benefits of enhanced homogeneity.

Conclusion

The synthesis and characterization of surfactant-based metal nonporous materials have proven to be a promising avenue for advancing various applications. The unique properties of these materials, stemming from the combination of surfactants and metals, offer enhanced functionalities in different fields. In conclusion, this research has contributed significantly to the understanding and utilization of surfactant-based metal nonporous materials, leading to notable advancements in diverse applications. he enhanced properties of these materials, coupled with the ability to tailor their structures, make them valuable contenders for addressing current challenges and advancing technologies in catalysis, sensing, energy, and beyond. Continued research in this area holds great promise for further breakthroughs and the development of innovative solutions to real-world problems.

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DOI: https://doi.org/10.15379/ijmst.v10i4.3663