Porous Microspheres: A Review of Synthesis Protocols, Physicochemical Characterization, and Multidisciplinary Applications.

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Abstract:

Significant attention is currently focused on porous microspheres within material science due to their extensive utility in sectors ranging from pharmaceuticals to environmental remediation. These spherical entities are distinguished by specific structural attributes, including high surface-to-volume ratios, tunable pore dimensions, and adaptable morphologies. This review presents a detailed examination of the primary materials used in their fabrication, along with an evaluation of established and emerging synthesis strategies. Although porous microspheres have been extensively reviewed within isolated disciplines, a comprehensive framework linking advanced, scalable fabrication techniques to cross-disciplinary applications is rarely presented. Therefore, a specific gap in the current literature is addressed by this review through the systematic correlation of emerging synthesis strategies, such as microfluidics and 3D printing, with their consequential impacts on structural tunability and multidisciplinary functionality. New perspectives on the transition from traditional bench-top synthesis to sustainable, large-scale production are contributed. In this manuscript, the synergistic roles played by organic, inorganic, and hybrid matrices in enhancing material stability are critically analyzed. Furthermore, the efficacy of modern preparation methodologies is assessed against traditional techniques, and essential characterization protocols are highlighted. Finally, the transformative deployment of these advanced microspheres in drug delivery, catalysis, and biosensing is explored, and critical bottlenecks associated with cost-efficiency and industrial scalability are discussed.

Keywords: *porous microspheres, synthesis protocols, physicochemical characterizations, drug delivery systems; environmental remediation*

Received: 26.12.2025 Accepted: 15.6.2026. Published: 30.6.2026

DOI: 10.59907/daujs.5.3.2026.548

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ISSN: 2815 - 5807

Introduction

Porous microspheres are defined as spherical micro-particles endowed with an internal porous network, characterized by regulated morphology and substantial surface area. (Lee & Patel, 2022) These structural features are exploited to facilitate high loading capacities, controlled release profiles, and selective molecular binding, rendering them indispensable in both industrial and scientific contexts. (H. Bai et al., 2025; Kang et al., 2013)

The performance of these materials is largely dictated by specific quantitative surface metrics rather than general structural characteristics. Broad specific surface areas, ranging from 50 to over 2500 m^2/g as determined by BET analysis, are frequently reported across different matrices, while internal pore sizes are typically tailored within the micro- (< 2 nm), meso- (2–50 nm), or macroporous (> 50 nm) regimes. Furthermore, particles with diameters ranging from 1 to 500 μm and total porosities between 30% and 90% are commonly synthesized to meet diverse application requirements. In fields such as catalysis and environmental cleanup, these extremely high surface areas maximize operational efficiency by significantly increasing the availability of active binding sites. In the pharmaceutical domain, the rate of release of encapsulated therapeutic agents is strictly governed by the precise engineering of the aforementioned pore dimensions. Beyond physicochemical parameters, practical manufacturing aspects, such as high production throughput, strict batch-to-batch reproducibility, and economic scalability, are continuously evaluated to ensure the successful industrial translation of these micro-entities. In applications such as catalysis and environmental cleanup, efficiency is maximized by the high surface area, which increases the availability of active sites. In the pharmaceutical domain, the rate at which encapsulated therapeutic agents are released is governed by the precise engineering of pore sizes. (H. Bai et al., 2025) Furthermore, in biosensing, the interaction between the microsphere and target analytes is optimized by controlling the shape of the particle. Consequently, these porous structures are viewed as critical solutions to complex challenges encountered in medicine, energy storage, and environmental science. (Gurung & Kakar, 2020)

Porous microspheres offer distinct advantages over other particulate systems, primarily due to their ability to tailor porosity and surface characteristics. This versatility ensures their effective adaptation across various fields requiring functional precision. (Cao et al., 2025; Y. Zhang et al., 2025) To systematically explore these capabilities, this review comprehensively summarizes the current state of the art in porous microspheres. Particular attention is directed toward recent advances concerning the foundational materials utilized, the evolution of sophisticated preparation methodologies, and the critical characterization techniques required for structural validation. Finally, the wide variety of multidisciplinary applications enabled by these micro-structures is thoroughly addressed, providing a cohesive perspective on their future technological trajectory.

Materials for fabrication

The synthesis of porous microspheres is achieved through the selection of specific materials, categorized into organic, inorganic, and hybrid groups, depending on the intended application.

Organic polymers

Organic materials, particularly polymers such as polylactic acid (PLA) and polystyrene, are frequently utilized. These polymers are valued for their flexibility, which allows for the easy modification of surface properties and pore architecture. (Gonzalez et al., 1999; Shi et al., 2018; H. Wang et al., 2006) Polylactic Acid (PLA): This polymer is predominantly selected for biomedical purposes. Due to its biocompatibility and the non-toxic nature of its degradation products, PLA is considered ideal for drug delivery systems requiring sustained release. (Abdul Hamid et al., 2018; Hyon, 2000) Polystyrene: Characterized by chemical resistance and mechanical toughness, polystyrene is employed in applications where durability is paramount, such as environmental remediation and biosensing. (G. Li et al., 2022; Ma et al., 2006) Functionalization is readily performed on these organic polymers to meet specific operational demands. (Moulay, 2018)

Inorganic matrices

Inorganic materials, including ceramics, silica, and carbon-based compounds, are chosen for their robustness.

- Ceramics: Excellent mechanical strength and thermal stability are provided by ceramics, making them suitable for harsh environments involving high temperatures or corrosive conditions. (Sivakumar et al., 2025)

- Silica: Microspheres composed of silica are favored for their exceptional porosity and chemical inertness, which are utilized effectively in adsorption and catalysis. (Wu et al., 2025)

- Carbon-based materials: Substances such as activated carbon and graphene are noted for their high surface area and electron transfer capabilities, which are used to enhance catalytic activity. (Sun et al., 2025)

Composite and hybrid systems

The beneficial properties of both organic and inorganic components are combined in composite materials. (Guo et al., 2025) For instance, mechanical strength and thermal stability are enhanced when organic polymers are reinforced with inorganic fillers, such as silica. In drug delivery contexts, silica-reinforced PLA microspheres are utilized to maintain biocompatibility while enhancing structural integrity. (Jing et al., 2025) Hybrid systems are

designed to enable specific interactions, such as the selective adsorption of pollutants, by leveraging the functional groups of organic polymers in conjunction with the high surface area of inorganic matrices. (He et al., 2025)

Synthesis methodologies

A variety of techniques are employed to manufacture porous microspheres, ranging from classical physical-chemical methods to advanced fabrication technologies.

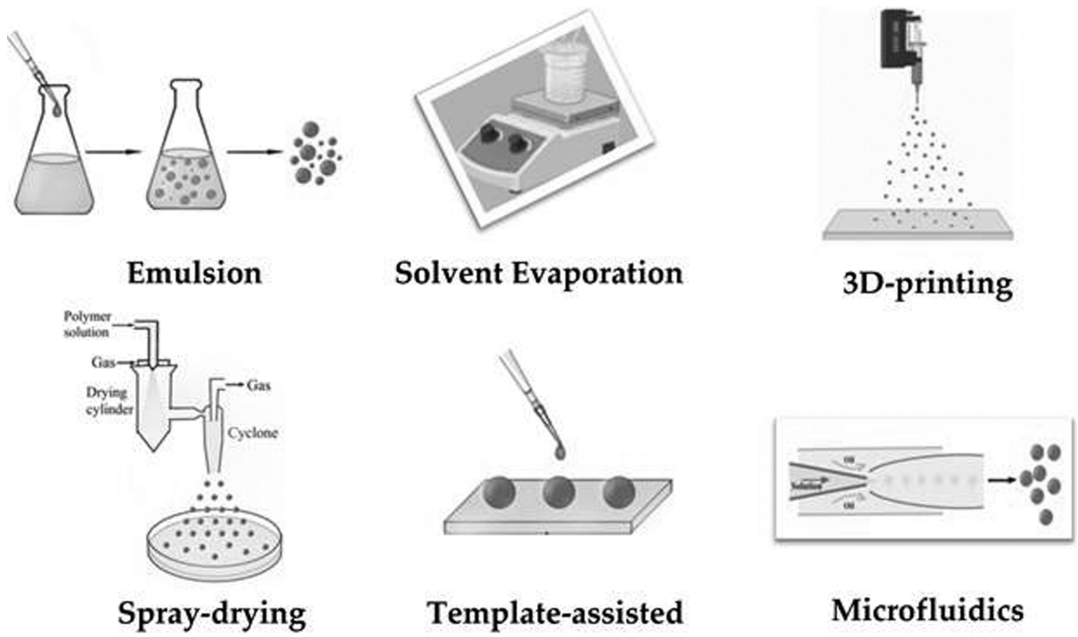

Figure 1. A variety of techniques are employed to manufacture porous microspheres.

Solvent evaporation and emulsion

Control over particle size and pore structure is reliably achieved through emulsion and solvent evaporation techniques. (Tuo et al., 2025)

- Solvent Evaporation: In this process, a polymer is dissolved in a volatile solvent. Solid microspheres form as the solvent evaporates. The resulting porosity is dictated by the rate of evaporation. (Sankeshi et al., 2025)

- Emulsion Technique: This involves the dispersion of a polymer solution into an immiscible liquid phase, stabilized by surfactants. Uniformity in particle shape and size is attained by adjusting stirring speeds and surfactant concentrations. (Ding et al., 2025)

These methods are widely regarded as versatile and reproducible for applications in drug delivery and catalysis.

Spray drying and freeze-drying

Thermal and cryogenic methods are utilized for specific material requirements.

- Spray drying: An atomized liquid feed is introduced into a hot gas stream, causing rapid solvent evaporation and the solidification of porous particles. This method is particularly applied to heat-sensitive compounds, such as proteins, to prevent degradation. (Q. Li et al., 2025)

- Freeze-drying (Lyophilization): This technique involves the freezing of a formulation followed by the removal of ice via vacuum sublimation. A structure with high pore volume is created as the ice crystals are eliminated, preserving the integrity of biological substances like vaccines. (Y. Bai et al., 2025)

Template-Assisted Synthesis

Specific pore architectures are generated using template-assisted synthesis. A matrix material is coated onto a template (solid, liquid, or gas), which is subsequently removed to reveal the porous structure. (Bhalala et al., 2025) While excellent control over pore distribution is offered by this method, challenges regarding the complete removal of template residues are acknowledged. (Jing et al., 2025)

Emerging Techniques

Modern technologies are revolutionizing production capabilities (Table 1).

- Microfluidics: uniform droplets and microspheres with precise pore diameters are generated through controlled fluid dynamics. (Huang et al., 2025)

- 3D printing: complex, customized inner structures are constructed using 3D printing, enhancing utility in tissue engineering. (Zeng et al., 2025)

- Green synthesis: environmentally friendly production is promoted by methods utilizing renewable materials and low-energy processes. (Abdullah et al., 2025)

Table 1. Comparative analysis of synthesis techniques (Cai et al., 2013).

Synthesis Method	Primary Mechanism	Key Advantages	Typical Limitations	Particle size
Solvent Evaporation	Dissolution of polymer followed by volatile solvent removal.	Simple, versatile control over particle size.	Residual solvent toxicity; long processing time.	0.1-1000 μm

Synthesis Method	Primary Mechanism	Key Advantages	Typical Limitations	Particle size
Spray Drying	Atomization of liquid feed into a hot gas stream.	Rapid, continuous processing; suitable for heat-sensitive drugs.	Particle size distribution can be broad; the equipment cost.	10-1000 μm
Freeze-Drying	Sublimation of ice crystals under vacuum.	Preserves biological activity (vaccines/ enzymes); high porosity.	High energy consumption; slow batch processing.	10-500 μm (dictated by initial formulation)
Template-Assisted	Coating a matrix over a sacrificial template.	Precise control over pore shape and architecture.	Difficulty in complete template removal; potential residue.	0.1-1000 μm (determined by template dimensions)
Microfluidics	Manipulation of fluids in micro-channels.	Extremely uniform particle size (monodispersity).	Low production throughput; complex setup.	1-1000 μm (highly tunable)
3D Printing	Layer-by-layer construction.	Ability to create complex, customized internal structures.	Resolution limits; material compatibility constraints.	> 50 μm (restricted by printer resolution)

Characterization techniques

To ensure the functionality of porous microspheres, rigorous analysis of their structural, thermal, and chemical properties is required.

Morphological and structural analysis

Porous materials are classified by the International Union of Pure and Applied Chemistry (IUPAC) based on pore size: microporous (< 2 nm), mesoporous (2-50 nm), and macroporous (> 50 nm). (Desoky et al., 2025)

- Microscopy: Detailed visualization of surface topology is provided by Scanning Electron Microscopy (SEM) (Figure 2a-b) and electron image (Figure 2c), while internal pore structures are examined using Transmission Electron Microscopy (TEM). (Zhao et al., 2025).

- Size Distribution: Particle stability and homogeneity are assessed using dynamic light scattering and laser diffraction techniques. (Maciejewska, 2025)

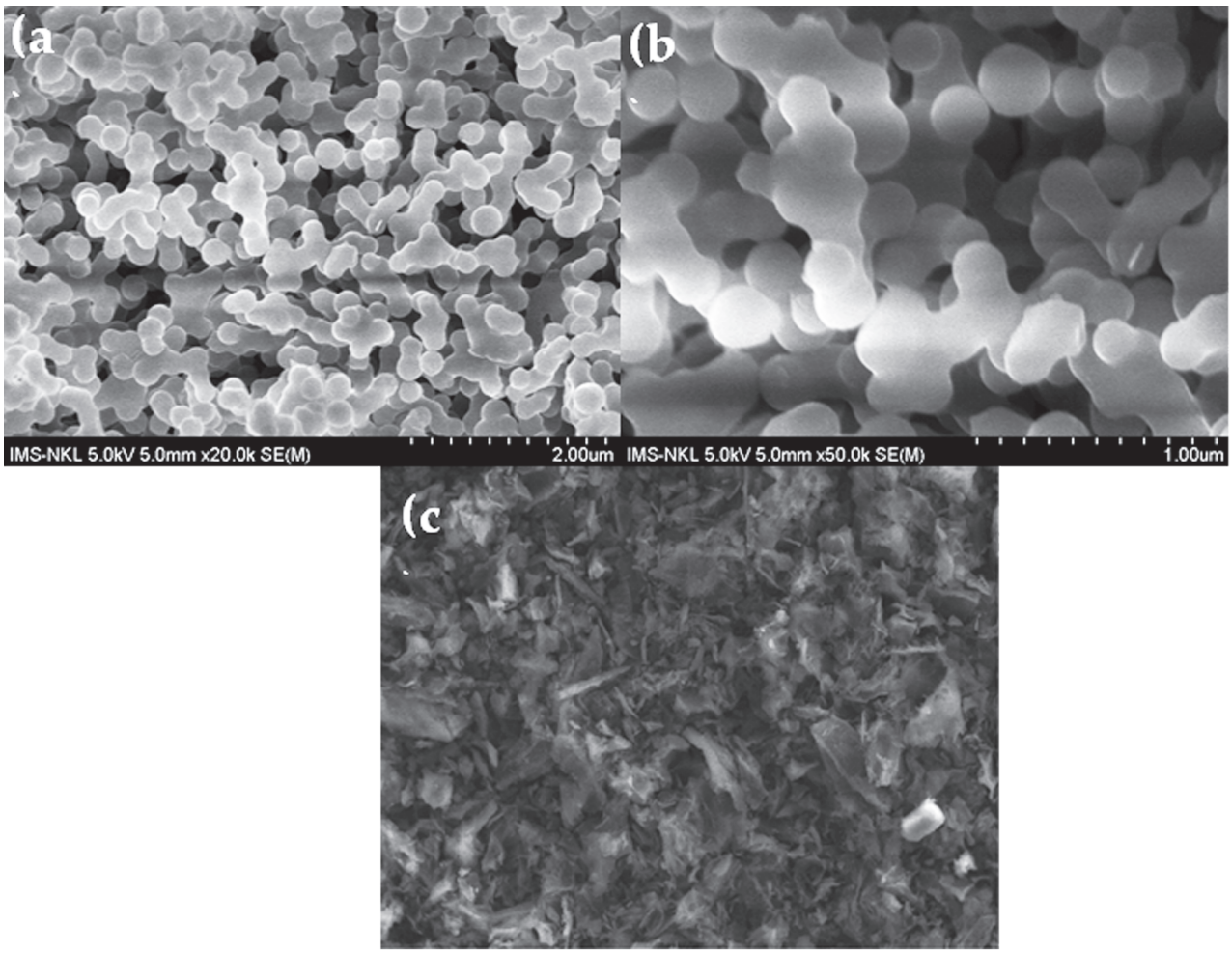

Figure 2. (a, b) The SEM and (c) electron images of carbon microspheres.

Porosity and surface area

Surface characteristics are quantified through Brunauer-Emmett-Teller (BET) analysis, where nitrogen gas adsorption is measured to determine the specific surface area. Additionally, pore volume and size distribution are determined by mercury intrusion porosimetry, which involves forcing mercury into the pores under pressure. These parameters are directly linked to adsorption capacity and catalytic efficiency. (T. Wang et al., 2025)

Thermal and mechanical stability

Material endurance is evaluated using thermal analysis.

- Thermogravimetric Analysis (TGA): Decomposition points and weight changes under thermal stress are monitored to assess stability. (Vindhyasarumi et al., 2025)

- Differential Scanning Calorimetry (DSC): Phase transitions, such as glass transition and melting, are identified by monitoring heat flow. (X. Li et al., 2024)

Mechanical strength is also tested to ensure the microspheres maintain integrity during handling and application. (Yang et al., 2024)

Chemical composition

The chemical nature of the microspheres is analyzed to confirm functionalization.

- FTIR: Molecular bonds are analyzed using Fourier Transform Infrared Spectroscopy to detect chemical modifications. (H. Li et al., 2025)

- EDX and XRD: Elemental composition is revealed by Energy-Dispersive X-ray (EDX) spectroscopy, while crystalline structures are determined by X-ray Diffraction (XRD). (J. Zhang et al., 2024)

Applications

Drug delivery systems

Targeted and regulated medication release is facilitated by porous microspheres, leading to reduced side effects and increased efficacy. (X. Zhang et al., 2024) Chemotherapeutic agents, such as doxorubicin, are encapsulated for direct delivery to tumor sites, thereby maximizing therapeutic effects while minimizing systemic toxicity. (Ding et al., 2025) Additionally, antigens are preserved and slowly released by these systems in vaccine formulations, enhancing long-term immune responses. (Dou et al., 2025)

Catalysis and adsorption

In the chemical industry, catalytic activity is significantly enhanced by the large surface area of these microspheres, which promotes interaction between reactants. (H. Bai et al., 2025; Gurung & Kakar, 2020) They function as effective catalyst supports in processes like hydrogenation. (Cao et al., 2025; Y. Zhang et al., 2025) Environmental remediation is also achieved through adsorption, where heavy metals and organic chemicals are captured from water sources using silica or activated carbon microspheres. (X. Zhang et al., 2024)

Biosensing and diagnostics

Biomolecules, such as antibodies and enzymes, are immobilized on the extensive surface of porous microspheres to detect disease biomarkers. (Y. Bai et al., 2025; Zeng et al., 2025) Specific interactions with viral proteins or cancer antigens are enabled by functionalized surfaces, permitting rapid diagnosis. (Gurung & Kakar, 2020; Y. Zhang et al., 2025) Furthermore, contrast agents are incorporated into these particles to improve medical imaging technologies, including CT and MRI scans. (H. Li et al., 2025; T. Wang et al., 2025)

Environmental and energy sectors

Beyond water treatment, carbon dioxide is trapped by the porous structure of these materials, contributing to climate change mitigation. (Lee & Patel, 2022; Sun et al., 2025) In the energy sector, electrode performance in batteries and fuel cells is improved by the high charge capacity and stability provided by porous microspheres. (Ding et al., 2025; Huang et al., 2025)

Conclusion and future perspectives

It is concluded that porous microspheres represent a versatile class of materials with significant potential across medicine, industry, and environmental science. (Bhalala et al., 2025; Desoky et al., 2025) It has been demonstrated that diverse materials—organic, inorganic, and composite, can be synthesized through a wide array of methods, from traditional emulsion to advanced 3D printing. (Dou et al., 2025; He et al., 2025) While their applicability is broad, challenges associated with scaling up production, managing costs, and ensuring long-term stability remain to be addressed. (Bhalala et al., 2025; X. Li et al., 2024)

Future research efforts are expected to focus on environmentally friendly "green" synthesis techniques to minimize ecological impact. (Dou et al., 2025) Furthermore, the performance of these materials is anticipated to be enhanced through integration with nanotechnology. (Cao et al., 2025; J. Zhang et al., 2024) Sustainable solutions to global issues, such as energy storage and healthcare delivery, are likely to be provided by the continued development and optimization of porous microspheres. (Dou et al., 2025; Yang et al., 2024)

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