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# To Study Mesoporous Silica Nanoparticles to Generate Functional Hybrid Silica Scaffolds with Controllable Hierarchical Porosity by Dynamic Templating

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**ABSTRACT**

**The emphasis of this review is therefore on non-ordered commercially available mesoporous silica and the progress that has been made in development of the use of these materials for improved dissolution rates in oral drug delivery. First, a thorough categorisation of the drug loading methods is presented, followed by discussion on the most important characteristics of solid dispersions (*i.e.*, physical state, stability, drug release). Finally, manufacturability and production of a final solid dosage form are considered. Mesoporous silica nanoparticle drug delivery systems are effective due to their structural properties, high drug loading capacity, biocompatibility, cost effective synthesis, and use of these nanomaterials as delivery systems for biological cells and targeted releases. In order to effectively transport and distribute extremely toxic drugs, such as chemotherapeutic agents for cancer treatment, MSNPs as promising nanocarriers. In conclusion, this review goes into great depth about the most recent developments in the synthesis and functionalization of MSNs. MSNPs are promising nanocarriers to efficiently transport and site-specifically deliver highly toxic drugs, like chemotherapeutic agents for cancer treatment. MSNs are efficiently transported site specific drug delivery of highly toxic drug such chemotherapeutic agents for effective cancer treatment. They have stimuli responsive drug releases so, it enhancing and minimizing the side effects of anti-cancer drugs in cancer therapy.**

**Keyword*: Physical State, Stability, Drug Release, Cancer Treatment, Biocompatibility***

## INTRODUCTION

Compared to "monomeric" building blocks, self-assembly of building blocks such colloids, molecules, polymers, etc. leads to the development of structures over a longer length scale. Ensembles of inorganic nanoparticles have inspired many researchers in recent years due to the potential to take advantage of individual nanoparticles' collective features and use them in functional devices.65. Nanoparticle assemblies can be utilised to enhance the mechanical characteristics of composite materials and enable the sequential or simultaneous execution of several functions. Both equilibrium and non-equilibrium processes can lead to the formation of self-assembling nanoparticulate formations. Inorganic crystals, organic crystals, and other surfactant/block copolymer microphase separated structures are typical instances of equilibrium self assembly.

Conversely, non-equilibrium systems include DNA replication, which deviates greatly from equilibrium. According to Whitesides et al., covalent or non-covalent forces including van der Waals, electrostatic, hydrogen bonding, and hydrophobic interactions can cause molecules to self-assemble. A number of other interactions, including capillarity and external fields like electric, magnetic, and gravitational fields, are crucial for the assembly of larger particles (meso or macroscopic objects).

Organising nanoparticles to create self-assembling structures has been used in a variety of fields, including photonics, sensing, electronics, and medication delivery, among others. High porosity is crucial for applications like catalysis, sensing, drug administration, sorption, separation, and tissue engineering in addition to having self-assembling nanostructures. Many applications require porosity at various length scales, such as micropores (less than 2 nm), mesopores (between 2 and 50 nm), or macropores (more than 50 nm). Since we illustrate how porous structures form by self-assembly in this thesis, we limit our discussion to literature relevant to these systems.

**Strategies to form porous structure:**
Because of its core scientific interest and several contemporary technological uses, the quest to create a material with well-defined pores has always been one of the most active fields in materials science engineering.76. One length scale or several length scales of porosity can be found in porous materials. If a material has pores with varying orders of length scales, such as micropores, mesopores, and macropores, it is regarded as hierarchical. Below are succinct summaries of the many approaches to producing hierarchical porous structure in this context.

**Template directed self assembly of colloids:**
Using templates is a popular technique for creating hierarchical porous materials. The structure-directing agents are often self-assembled molecular aggregates or supramolecular assemblies. In template-assisted self assembly, 1D, 2D, or 3D substrates with colloidal particle localisation sites are used. Thus, complex structures determined by the template's shape are created by templated assembly.77 The following succinctly describes the creation of organised materials using templates: (a) the template is combined with colloidal particles or precursor solution; (b) the particles occupy the voids or empty spaces on the template's surface and arrange themselves to form an inverse structure; and (c) the templates are eventually removed after fixation, for instance by chemically cross-linking the colloidal structure. In general, there are two types of templates: soft and hard.

Soft templates are usually organic compounds that can be eliminated by mild dissolve conditions or regulated heat treatment. Surfactant micelles, amphiphilic copolymers, and biological molecules such as proteins, viruses, and DNA are examples of these. After self-organization, these templates typically result in mesopores in the material. However, some large-sized materials, like colloidal crystals, inorganic salt and ice crystals, and mesoporous silica materials like MCM-41, MCM-48, SBA-15, and porous polycarbonate, can be regarded as hard templates and are added during the synthesis to guide the formation of macroporous structures. To get rid of these rigid templates and create highly organised inverse macroporous structures, harsh acid or alkali leaching procedures are typically needed. Spray drying, dynamic templating utilising breath figures, polymerization-induced phase separation, and layer-by-layer assembly are some of the several techniques for organising colloids using templates that have been documented. Emulsion droplets, vesicles, bubbles, colloidal crystals, polymer foams, membranes, polymer gels, biopolymers, biological templates, and liquid crystals are some of the several template types utilised to assemble nanoparticles. Inverse replica materials have been created using colloidal crystals as the template. Initially, precursor solution or nanoparticles are combined with colloidal crystals. When crystals are removed, hollow sphere arrays of nanoparticles or three-dimensionally organised macroporous (3DOM) materials are created. Inverse inorganic structures with various morphologies can be created using a variety of naturally occurring, low-cost, and environmentally safe biological templates, including bacterial threads, echinoid skeletal plates, egg-shell membranes, insect wings, pollen grains, plant leaves, and wood celluloses.

## RESEARCH METHODOLOGY

Reactants can also be contained within nanoscale domains using surfactant mesophases as templates. Space is divided into hydrophilic and hydrophobic areas by the liquid crystalline mesophase. As a result, the hydrophilic and hydrophobic reactants separate into their respective areas. According to Attard et al., the condensation of tetramethyl orthosilicate (TMOS) produces silica when non-ionic surfactant mesophase is used as a template. A hexagonal mesophase (H1) was created using a combination of C12E8/H2O/TMOS; the TMOS was hydrolysed and condensed within this under acidic conditions to generate silica. After the surfactant was removed from this composite by calcining it, an ordered hexagonal silica structure with nm-sized pores was produced. The lamellar phase of another surfactant, C16E8, was used to create similarly structured silica structures. The use of liquid crystals of surfactant assemblies to create different types of porous silica structures has now advanced significantly. Guruswamy et al. recently showed how to use the non-ionic surfactant C12E9. as a template to create macroporous silica materials. A network of nanoparticle strands is created by assembling the silica nanoparticles via dynamic templating of surfactant hexagonal domains. When the surfactant matrix cools into the hexagonal phase, dispersed particles (>10 nm) form networks regardless of particle chemistry. It is possible to reach a macroporous substance once the surfactant has been removed.

**Multiple templating approach to generate hierarchical porous materials:**
Multiple structure guiding templates can be used to create hierarchically porous materials by combining bigger pores with micro or mesopores. Mesoporous inorganics are condensed around macroscopic templates in a popular method for creating hierarchically porous materials. For instance, mesoporous silica was synthesised over sacrificial polymer lattices in early attempts to create meso/macroporous materials. The breath figure approach or the synthesis of micro or mesoporous structures surrounding polymer colloidal crystals have produced ordered macroporous structures. Using salt crystals as templates or in conjunction with top-down methods like micromolding, more intricate morphologies, such as non-spherical macropores, have been created. Hierarchically porous materials have also been synthesised using emulsions and foams as templates. Additionally, controlled phase separation of porogens to produce macroporosity has been reported.

It is also possible to create hierarchically porous materials by assembling prefabricated micro- or mesoporous particles to create macroporosity. For instance, surface-functionalized mesoporous silica particles were cross-linked around triblock copolymer assemblies to create meso/macroporous materials. Hierarchically porous materials have also been created by carefully freezing silica microsphere dispersions. It was also recently reported that functionalised mesoporous silica particles were "protected" by embedding polymer in a porous silica matrix.

## RESULT AND DATA ANALYSIS

The potential for covalent grafting of molecular systems on its surface and its amorphous nature, which makes it a suitable material to cover any metal or metal oxide surface to disclose a core-shell architecture, were two factors in the decision to use silica as a matrix. The catalytic activity of a single homogeneous catalyst that was immobilised on the surface of silica nanoparticles was covered in the previous chapter. It would be preferable for multistep organic synthesis if a single catalyst could perform two successive reactions.2 If such a multifunctional catalyst could be created, the difficulty of separating heterogeneous catalyst after each reaction would be reduced.3 Additionally, the benefit of such a system is that the time and yield losses related to the isolation and purification of intermediates in multistep sequences would be eliminated by numerous catalysts working in tandem. The development of such contemporaneous tandem catalytic systems has so received a great deal of attention. The development of a composite catalyst that can perform two successive reactions has been the primary focus of this chapter.

Multiple catalysts must be immobilised in order to produce such a composite catalyst for two simultaneous processes.1 The industrialisation of homogeneous catalysts depends on catalyst immobilisation.2 However, precisely anchoring various catalysts on the firm foundation is difficult. The reaction sequence may also be hampered by the proximity of the catalysts' active regions. Porous matrices, which function as a nano reactor, have been widely used to address these problems. Reactant molecules can be transported by grafting a catalyst into a porous support's pore. Mesoporous silica materials with well-ordered arrays of uniform nanometer-sized channel pores5 and controlled porosity4 have been widely employed as hard templates for the immobilisation of homogeneous catalysts, among other solid supports. In such a setup, two distinct catalysts—one inside the pore and the other on the particle surface—could be rendered immobile.

Enzymes or metal nanoparticles are known to catalyse a wide range of industrially significant processes.6-8 However, a significant obstacle to its use in a variety of industrial processes is the catalytic component's high cost and scarcity.9 Even in the field of nanoparticles, several structures have been proposed and tested, including carbon-supported catalysts (17), polymer nano conjugates (10–13), and core-shell particles placed inside porous matrices (14–16). Mesoporous silica-coated metal nanoparticles18 have already been employed in numerous literature studies as a nano-reactor19 for a variety of catalytic reactions, such as catalytic reductions of p-nitrophenol20,21 or photo-catalytic activities in the degradation of phenols and Cr (VI). Enzyme and transition metal catalysis appears to have gotten far less attention, despite the fact that the majority of efforts have been focused on developing tandem catalytic systems based on organometallic complexes.

According to this theory, the design and synthesis of enzyme-grafted mesoporous silica, which is then coated on the surface of a metal nanoparticle, is an attractive concept because it allows for the execution of a multistep reaction sequence that calls for either independent or cooperative catalytic performance of the metal and enzyme. There are numerous examples of bifunctional mesoporous material catalysts in the literature that combine two distinct organic functional groups, such as amines and sulphonic acid or bronsted acid and bronsted base, to perform two sequential reactions.22–24 To the best of our knowledge, however, no reports have been made of the immobilisation of enzymes on the surface of mesoporous silica coated on nanoparticles so that two simultaneous reactions can be carried out. As one might anticipate, there are numerous significant obstacles in the way of developing such complex systems. The first step is to combine the metal catalyst and the enzyme into a matrix so that the metal catalyst does not interact with the enzyme's many functional groups, denaturing it and causing activity loss.25. Second, in order to prevent the metal and enzyme from leaching into the reaction mixture, we should be able to recover both the metal nanoparticles and the enzyme. In order to prevent aggregation during the process, the metal should also have the proper surface passivation.26. Lastly, the substrates should have easy access to both the enzyme and the nanoparticles. Incorporating the metal catalyst and enzyme in a heterogeneous support12,27,28 such that they are accessible to small organic molecules but do not interact with one another would be one method to get around all of these problems.

The design and production of mesoporous silica nanoparticles (MSN) coated on gold nanoparticles in a core-shell structure with an enzyme grafted on the exterior silica surface are presented here. Because of this design, these two catalysts can work together or separately to carry out a two-step reaction without interfering with one another. This hybrid material is appealing due to a number of characteristics. (i) Enzymes can be readily grafted to the exterior surface of Au nanoparticles encapsulated in MSN using a straightforward one-pot process. (ii) This hybrid material enables two simultaneous reactions to occur in parallel, with the first reaction's result serving as the second reaction's substrate. This is comparable to a "relay" race in which the first competitor's performance determines the second's. (iii) The hybrid MSN can be separated by straightforward centrifugation at the conclusion of the procedure and then used

again. The enzyme glucosidase, which was anchored on the exterior surface of MSN, hydrolysed 4-nitrophenyl-β-glucopyranoside in the first phase of a cascade reaction used as a proof of concept. The next stage involves the release of 4-nitrophenol from the previous reaction diffusing into the MSN pores, where gold nanoparticles in the presence of NaBH4.4,14 convert it to the corresponding amine. Additionally included are the specific kinetics of both reaction stages.

**3.2 Experimental section:**

**3.2.1 Materials:**

Tetraethyl orthosilicate (TEOS), cetyltrimetmylammonium bromide (CTAB), -glucosidase, 4-nitrophenyl-glucopyranoside, NaBH4, HAuCl4, and (3-glycidyloxypropyl) trimethoxysilane were acquired from Sigma Aldrich and utilised exactly as supplied. The remaining solvents were acquired from Merck in India and utilised exactly as supplied.

**3.2.2 Synthesis:**

**3.2.2.1 Preparation of Au nanoparticles:**

The borohydride reduction process was used to create Au nanoparticles.29 500 mL of 10-4 M HAuCl4 was made. 50 mg of NaBH4 was gradually added to this mixture. For full reduction, the reaction mixture was left overnight.

**3.2.2.2 Preparation of Au@mSiO2 particles:**

Twenty millilitres of the previously prepared Au sol were transferred into a round-bottom flask. In that solution, cetyltrimethylammonium bromide (CTAB, 0.007 mg, 10-3mol) was dissolved and agitated for 30 minutes. After adding NaOH (aq) (2.00 M, 100 uL) to the CTAB solution, tetraethyl orthosilicate (20 L, 0.091 mmol) was added dropwise. After two hours of stirring, the reaction mixture was centrifuged twice using ethanol. The particles were refluxed in ethanol for a whole nite in order to totally eliminate the CTAB. These particles were then preserved as a dispersion in ethanol for later use after the solution was centrifuged once more.

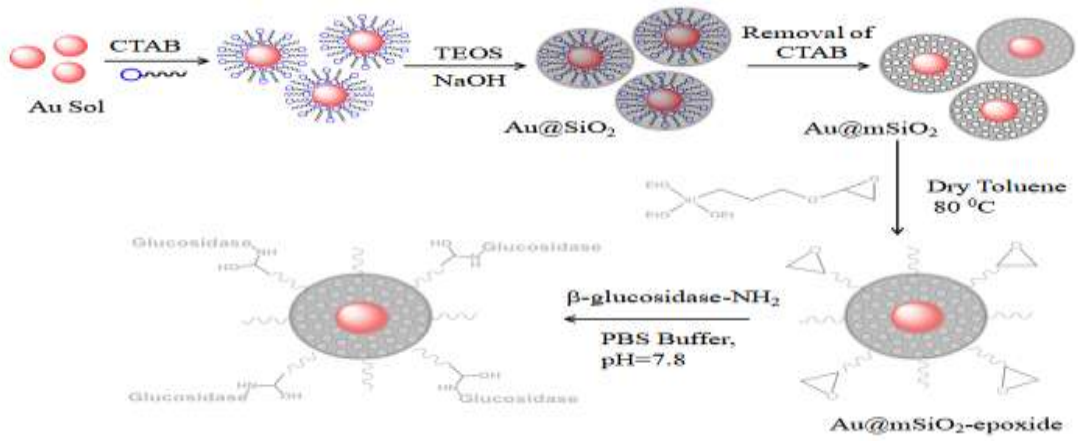
**3.2.2.3 Grafting of epoxide groups onto Au@mSiO2:**

Epoxide functional group grafting was carried out in accordance with a previously published protocol.27. A typical reaction involved dispersing 15 mg of Au@mSiO2 nanoparticles in 50 mL of dry toluene. After adding (3-glycidyloxypropyl)trimethoxysilane (10 L, 0.04 mmol) to this dispersion, the reaction liquid was heated to 85 C for 12 hours. The unreacted (3-glycidyloxypropyl)trimethoxysilane was eliminated by centrifuging and repeatedly washing the epoxide grafted core shell nanoparticles with ethanol after the reaction was finished.

**3.2.2.4 Immobilization of enzyme:**

Covalent enzyme anchoring was performed using previously published techniques.27,30,31 The first epoxide-grafted Au@mSiO2 nanoparticles (3 mg) were sonicated to disperse them in PBS buffer (pH = 7.4, 0.1 mM). After that, these were incubated at 4 °C for a nite with beta-glucosidase (2 mg, 1.48 X 10-8 mol). After centrifugation was used to separate the resultant hybrid nanoparticles, Au@mSiO2@glucosidase, the unreacted and absorbed enzyme was repeatedly rinsed with PBS buffer. The particles were employed as a stock after being distributed in 4 millilitres of PBS buffer. Nanoparticles coupled with enzymes were kept at 4°C. UV-Vis spectroscopy was used to measure the amount of enzyme immobilised on Au@mSiO2@glucosidase nanoparticles by tracking the absorbance at 280 nm. For every measurement, unfunctionalized Au@mSiO2 nanoparticles served as a blank.

Three phases make up our method for creating enzyme-grafted hybrid material (Scheme 3.1). The creation of Au core mesoporous silica shell (Au@mSiO2) nanoparticles was the initial stage in this endeavour. In the second stage, conventional silane chemistries were used to graft epoxide onto the exterior of these particles. In order to create the hybrid catalyst Au@mSiO2@glucosidase, -glucosidase was finally immobilised via the ring opening of epoxide by the NH2 groups of lysine contained in -glucosidase.



**Scheme 3.1: Schematic illustration of the synthetic procedure for gold nanocrystal /mesoporous silica core–shell NPs.**

Preformed Au nanoparticles were used in the sol-gel process to create the core shell nanoparticles (Au@mSiO2). Borohydride reduction of the metal precursor (HAuCl4) was used to create the Au nanoparticles. We employed the standard Stober Method to create the silica shell on Au nanoparticles. Cetyltrimethylammonium bromide (CTAB), an ionic surfactant, was applied to the nanoparticles. In addition to acting as an organic template for the sol-gel reaction's mesopore production, CTAB stabilises Au nanocrystals in the aqueous phase.32. Transmission electron microscopy was used to characterise the resultant core-shell nanoparticles after they had been separated by centrifugation and redispersed in ethanol. The particles were found to be widely distributed in water following silica coating. In order to gather Au@mSiO2 particles, the CTAB templates from the as-synthesised material were finally eliminated by heating them at reflux in ethanol.

Using well-established silane chemistry, the epoxide functional group was grafted. (3-glycidyloxypropyl) trimethoxysilane was applied to Au@mSiO2 nanoparticles in dry toluene for 16 hours at 80 °C. The substance was examined using TGA analysis in the presence of air and infrared spectroscopy following three ethanol washes.

The epoxy-grafted Au@mSiO2 nanoparticles were reacted with β-glucosidase in PBS buffer (pH=7.8, 0.1 mM) so that the amino groups of lysine in β-glucosidase cause the epoxy group on the surface of Au@mSiO2 to ring-open, covalently immobilising the enzyme on the surface of the nanoparticle (Au@mSiO2@glucosidase). Following the completion of the reaction, the material was repeatedly cleaned with a buffer system until the enzyme's distinctive UV absorption was absent from the supernatant. To ensure that no enzymes were electrostatically bound to the surface of Au@mSiO2 nanoparticles, control experiments were carried out. Initially, the β-glucosidase grafted Au@mSiO2 was cleaned with NaCl (0.1M) to weaken the electrostatic attraction between the surface of the nanoparticles and non-covalently bound enzymes, making them easier to remove. We deduce that our synthetic process results in the covalent attachment of enzyme to the nanoparticle surface and that any non-covalently attached enzymes are washed away during the purifying process because no enzymes were seen in the supernatant during these washings. Second, when β-glucosidase was incubated on bare Au@mSiO2 (particles without the epoxy group grafted on them), the enzyme did not adhere to the surface of the nanoparticles.

## CONCLUSION

(A) To enable silica to bind Au(III) ions, a functional group was formed on its surface using "Click Chemistry." In Hashmi's phenol synthesis, a reaction where Au(III) activates a terminal alkynes to isomerise o-alkynylfurans to phenols, the resultant composite material has demonstrated catalytic activity. This procedure is easily adaptable to other catalysts based on molecules or metals.

(B) The creation of a composite material that allows for the execution of two tandem reactions was also taken into consideration. The goal was accomplished by immobilising the glucosidase enzyme on an Au core and mesoporous silica shell type architecture. A two-step sequential reaction, in which a surface-bound enzyme catalyses the first reaction and metal nanoparticles trigger the second reaction on the product released from the first reaction, was made possible by the design's originality.

(C) The following chapter reports excellent control and tenability of the hydrophobicity and hydrophilicity of the mesoporous silica nanoparticles' inner and outer surfaces. Mesoporous silica nanoparticles' outer surface was grafted with a hydrophilic PEG group, while their inner surface was functionalised with methyl groups.

(D) Dynamic templating of liquid crystalline mesophases was used to build the mesoporous silica nanoparticles as a step ahead in our effort to replicate the "living" system. Scaffold macroporosity and nanoparticle mesoporosity might be controlled. This method has demonstrated the spatial tuning of macroporosity in a single sample. These scaffolds are also functional. Continuous flow reactors can also make advantage of this monolithic construction.

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