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Developing Hybrid and Functional Materials with Sol-gel Chemistry – A Review

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ABSTRACT

The sol-gel technique is a robust and chemically adaptable synthesis route for producing sophisticated inorganic, organic-inorganic hybrid, and composite materials. The sol-gel process uses hydrolysis and polycondensation reactions of metal alkoxide or salt precursors to generate nanostructured materials with controlled porosity, shape, and surface functionality. This bottom-up technique enables the synthesis of a wide range of material shapes, including thin films, aerogels, xerogels, fibers, coatings, and monolithic structures, under relatively mild circumstances. The incorporation of templating techniques and hybridization schemes has increased the adaptability of sol-gel produced materials, improving their mechanical and functional qualities. Applications include a wide range of fields, such as optical and electronic devices, energy storage systems, environmental remediation (e.g., removal of heavy metals and BPA), gas separation membranes, and biomedical applications for drug delivery and tissue engineering. The intrinsic capacity of sol-gel process to tune structural, chemical, and mechanical properties at the molecular level makes it a key component in the design and fabrication of next-generation functional materials. The mild synthesis conditions and adjustable nanostructures help the structures overcome important constraints in the biomedical and energy storage domains. Sol-gel-derived oxides such as TiO_2 and RuO_2 provide high surface area, enhanced cyclic stability, and improved ion diffusion in batteries and supercapacitors, achieving capacitances exceeding 700 F/g and capacities up to 190 mAh/g. In biomedical applications, sol-gel bioactive glasses promote rapid bone regeneration due to their high porosity and reactivity, while sol-gel silica nanoparticles enable controlled drug delivery with high loading efficiency. Sol-gel synthesis, in contrast to conventional techniques, permits the incorporation of delicate biomolecules without causing thermal degradation. Sol-gel is a potent platform for next-generation functional materials because of its adaptability.

Keywords: Sol-gel process; Organic-inorganic hybrid; Biomedical applications; Synthesis process.

1. INTRODUCTION

The demand for nanomaterials has increased significantly due to their unique properties and applications in various sectors, including electronics, energy, environmental cleanup, and healthcare. Among the different methods employed to produce nanomaterials, the sol-gel technique has emerged as one of the most promising due to its versatility, accuracy, and accessibility. Alternative techniques, such as mechanical alloying, electrochemical deposition, plasma-enhanced processes, and vapor-phase synthesis, are also commonly employed; however, each has drawbacks related to expense, complexity, or environmental consequences (Takahashi *et al.* 2006a). The sol-gel method stands out because it enables the formation of advanced materials under mild conditions, typically at or around room temperature. This low-temperature method enables enhanced control over material characteristics, such as composition, surface area, and microstructure (Tohge *et al.* 1994). Consequently, the sol-gel technique is ideal for generating high-purity substances in various forms, including thin films, fibers, powders, and nanoparticles. Its adaptability has led to applications in optics, electronics, energy systems, and biomedicine (Ying *et al.* 2006). A highly appealing aspect of the sol-gel method is its capacity to customize materials on a molecular scale. Researchers can carefully modify characteristics such as porosity, thermal stability, and mechanical strength by altering processing parameters like precursor concentration, water-to-alkoxide ratios, pH, and reaction duration (Kawamura *et al.* 2010; Kotani *et al.* 2000). For example, materials produced through sol-gel techniques frequently exhibit mesoporous configurations, improving their efficacy in catalysis, adsorption, and drug delivery.

This process typically involves two fundamental stages: (i) the preparation of a solution (sol), and (ii) the transition of the solution to a gel. In the first step, a metal alkoxide or inorganic salt is dissolved in a suitable solvent, followed by controlled hydrolysis and condensation reactions that lead to a colloidal suspension. During the reaction, the particles grow and aggregate to form a three-dimensional gel network (Matsuda *et al.* 2006). The use of acidic or basic catalysts tunes the rate of these reactions and influences the final network structure. The kinetics of the response must therefore be carefully controlled to obtain a successful

gelation. For example, a slow condensation rate helps to form a more homogeneous gel structure by preventing premature particle aggregation (Chen, 2001). This ability to control the way the materials develop structurally is a particular advantage of the sol-gel process over traditional solid-state synthesis processes, which typically lack this level of molecular precision.

Additionally, the sol-gel process allows for the preparation of organic-inorganic hybrid materials in which both components are homogeneously distributed within a single, synergistic structure. Such materials can be used where both mechanical properties and chemical properties are required, and they are generally cheaper to produce than vacuum-produced materials (Tadanaga *et al.* 2008). The capacity to produce films, coatings, and devices composed of well-mixed multiple components is another advantage of the sol-gel method. Control of the hydrolysis and condensation rates makes possible the production of materials with controlled particle sizes, interconnected pore structures, and given textures, all of which are relevant for high-performance applications (Djuriić *et al.* 2010; Ebrahimifard *et al.* 2014).

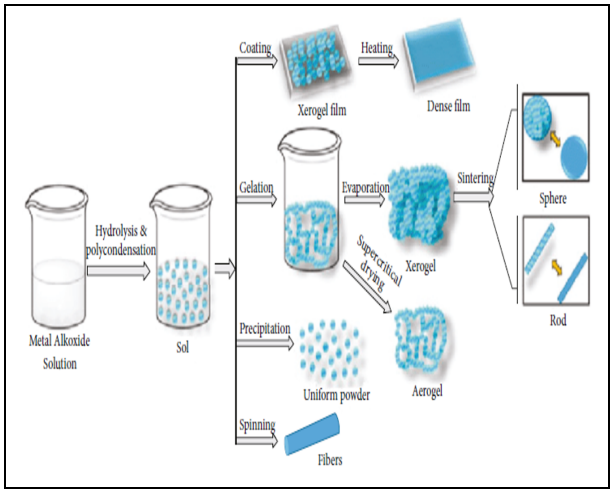


Fig. 1: The sol-gel process (Kumar *et al.* 2015; Ibrahim *et al.* 2013)

2. SOL-GEL

A liquid with small particles suspended in it is called a colloidal solution. Gelation, or the change from a sol to a gel, is the outcome of the gradual aggregation of these particles, which creates a 3D complex gel network in Fig. 2. One of the metal alkoxide precursors reacts with water after having been mechanically combined with the other. As a result of this reaction (Tadanaga *et al.* 2004; Tadanaga, 2013), a colloidal solution (sol) is formed through hydrolysis and polycondensation. Through a gelation process, a three-dimensional network is formed. This happens through the processes of hydrolysis and polycondensation, which turn the sol into a gel.

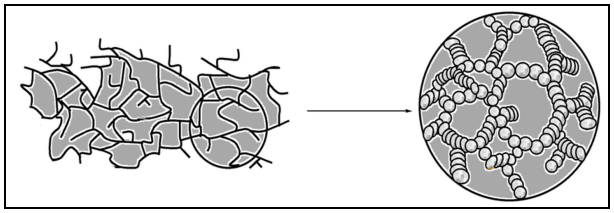


Fig. 2: Gelation to form a linked 3-D network (Kumar *et al.* 2015)

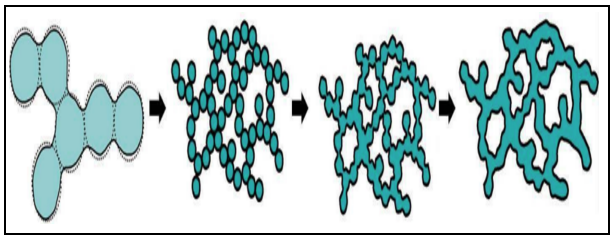


Fig. 3: Aging process (Kumar *et al.* 2015)

3. THE STEPS IN THE SOL-GEL METHOD

Mixing: In this step of the sol-gel process, a colloidal solution is formed by physically combining colloidal particles with water as a solvent, while maintaining pH control to prevent precipitation. After hydrolysis and polycondensation with water, the metal alkoxide precursor ($M(OR)_4$) produces a colloidal dispersion of ultrafine particles (1-2 nm). A three-dimensional network of equivalent inorganic oxide is formed over time by these particles.

Casting: The sol's low viscosity makes it simple to shape or cast into molds. The right mold must be used to keep the gel from adhering to the casting container.

Gelation: Three-dimensional networks are formed from condensed silica and colloidal particles as the process goes on; the gelation process and particle size determine the gel's final characteristics. Due to an increase in viscosity during gelation, the gel takes on the shape of the casting container. As gelation proceeds, fibers are spun together over time as viscosity is meticulously regulated. The agglomeration of colloidal particles results from interactions between their components, which are often electrical.

Aging: The term syneresis refers to this phase of the sol-gel process. The aging process of the cast object can take anywhere from a few hours to many days. Throughout this time, the gel network undergoes reprecipitation, and polycondensation proceeds within the solution in which the gel is submerged. As a result of this process, the interparticle necks become thicker, and the porosity decreases, which in turn increases the gel's strength. This enhances the endurance of the finished product by making the gel more resistant to breaking during the drying process, as shown in Fig. 3.

Drying: Excess solvent is eliminated from the intricate gel network during drying. In cases when the pores are tiny, drying can lead to the development of substantial capillary tensions. Cracking of the gel could result from these forces. It is crucial to regulate the drying process by lowering the liquid surface to avoid cracking. This can be accomplished by employing hypercritical evaporation, which prevents the solid and fluid components from interacting, adding surfactants, or removing tiny holes. When the gel dries, it transforms into an aerogel with a low density. The aerogel provides superior thermal insulation when evacuated and sandwiched between glass plates.

Densification: Following drying, the gel is heated to high temperatures in a furnace to encourage densification. This procedure compacts the material and removes any remaining organic matter, thereby strengthening the structure. The surface area, pore size, and interpore connection of the gel affect the necessary temperature level.

3.1 Specific Challenges or Limitations in Existing Synthesis Techniques

1. **Inadequate Control Over Composition and Microstructure:** Particle size, porosity, and homogeneity are not precisely controlled by traditional methods. At the nano- and meso-scale, sol-gel allows for tunable porosity and uniform element distribution by facilitating molecular-level mixing.

2. **Scalability and Versatility:** For thin films, coatings, or nanostructures, mechanical or thermal approaches frequently lack flexibility. With straightforward processing techniques like dip-coating or spin-coating, sol-gel can form powders, monoliths, films, and fibers.

3. **Limited Compatibility with Organic/Inorganic Hybrids:** The majority of traditional techniques are tailored for either inorganic or organic materials. Co-assembly of both is made possible by sol-gel, resulting in organic–inorganic hybrid materials with complementary qualities (such as flexibility and hardness).

4. **High Processing Temperatures:** High temperatures (>1000°C) are frequently needed for conventional ceramic or glass synthesis, which can break down delicate components. Because sol-gel synthesis takes place at room temperature or lower, it is perfect for creating hybrid materials or embedding biomolecules.

5. **Ineffective Doping or Functionalization:** Melt-based or solid-state techniques frequently struggle to evenly dope functional ions or molecules into a matrix. Sol-gel produces more stable and useful materials by enabling surface functionalization and in-situ doping during gelation.

3.2 Precursors for Sol-gel Processing

The precursor is the raw ingredient used in the sol-gel process.

- Precursors must dissolve in the reaction media.
- They must possess sufficient reactivity to engage in the gel-forming process.

As long as they are soluble in the right solvents, various compounds, including salts, oxides, hydroxides, complexes, alkoxides, acrylates, and amines, can be used as precursors in the Sol-Gel process. The most widely used of these are alkoxides because of their favorable reactivity and commercial availability. Selecting the right precursor for a particular application can be challenging because its reactivity depends on the chemical nature of the compound and the reaction conditions. Silicon is particularly good at forming strong gel networks. So, expensive metal alkoxides can frequently be substituted with less expensive silicon-based precursors, such as TEOS, TMOS, or water-soluble sodium silicate (Na_2SiO_3) for Sol-Gel processing.

3.3 Techniques for Turning Wet Gel into Aerogel

3.3.1 Drying Techniques

Solvent evaporation can be used to dry organic gels made in organic solvents at room pressure without seriously reducing their size or destroying their structural integrity. Surfactants are frequently used in these circumstances to help lower capillary pressure and lower the chance of cracking. Besides ambient drying, various techniques comprise other standard drying processes that aim to efficiently extract the solvent while preserving the gel structure.

- Chemical additives, including glycerol, formaldehyde, oxalic acid, tetramethylammonium hydroxide, and others, are used to regulate the rate of drying and reduce internal stresses in the gel.
- Ambient pressure drying does not require specialized equipment, as it facilitates solvent evaporation and is suitable for industrial-scale applications.
- Freeze-drying, also known as lyophilization, involves freezing the liquid aqueous portion of the gel and subsequently sublimating it to leave behind a lightweight, porous solid referred to as a cryogel.

Materials called xylogels are formed by drying in the open air. The primary distinction between them and aerogels lies in their pore structure, despite certain similarities. Although xylogels have fewer and smaller pores due to the limitations of traditional drying methods, aerogels, which are dried using supercritical drying techniques, typically have a greater number of larger cavities.

3.3.2 Drying by Supercritical Method

The process of supercritical drying involves heating wet gels under regulated pressure and temperature in a sealed chamber. These parameters are precisely calibrated to fall inside the solvent's critical point, which is trapped in the gel's pores. It can be extracted as a gas at this point because the solvent changes from a liquid to a gas without going through a separate liquid-vapor phase. The density and thermal conductivity of supercritical fluids are similar to those of liquids, yet they disperse like gases. The low surface tension of the fluids helps keep materials like porous structure of the aerogels intact by preventing collapse during drying. The characteristics of a few common supercritical fluids are displayed in Table 1. Fig. 4a illustrates the use of pressure-temperature diagrams to determine the critical point, and Fig. 4b displays the diagram of a supercritical drying autoclave.

Table 1. Some typical supercritical fluids and their characteristics

S. No.	Compound	Critical Temperature (°C)	Critical Pressure (bar)
1	Water	374.15	218.3
2	Ethylene	9.21	49.7
3	Nitrous oxide	36.5	71.7
4	Ammonia	132.4	112.5
5	Carbon dioxide	31.3	72.9
6	Methanol	240.5	78.9
7	Xenon	16.6	57.6
8	Propane	96.67	41.9
9	Krypton	-63.8	54.3
10	Pentane	196.6	33.3

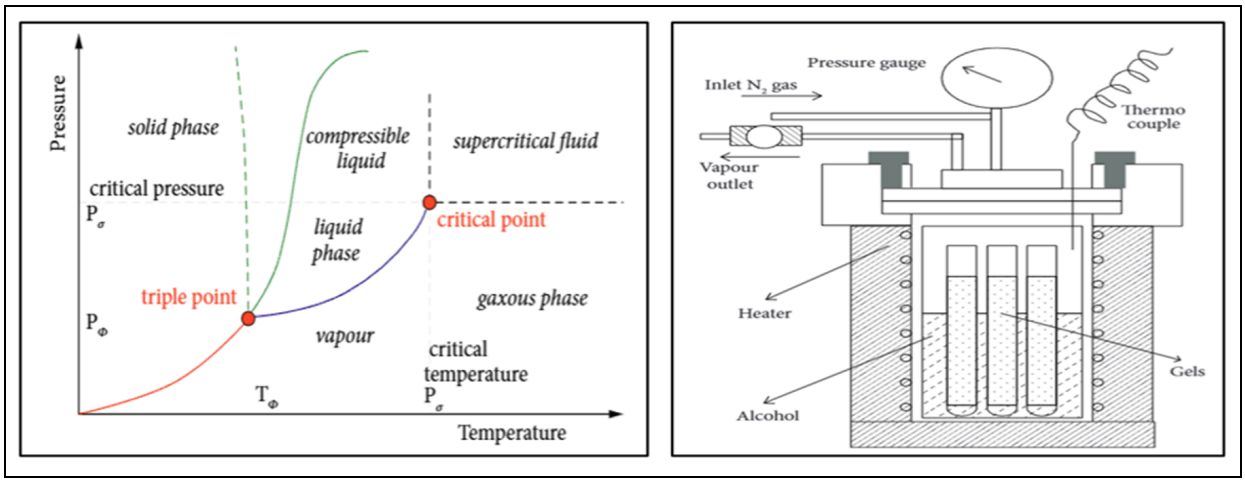


Fig. 4: (a) Methods for determining the critical point with pressure-temperature diagrams and (b) a schematic of an autoclave for supercritical drying

4. TYPES OF AEROGELS

Based on the chemical composition of their gel networks, aerogels can be categorized into four primary types: inorganic, organic, carbon-based, and hybrid.

- **Inorganic Aerogels:** These are usually made in an acidic environment. Examples of these include silica and alumina-based aerogels. Numerous metals and semiconductor oxides, including titanium, zirconium, tin, vanadium, iron, and niobium, can also be used to form these materials. Due to their ceramic composition, inorganic aerogels are often brittle despite their versatility.
- **Organic Aerogels:** Alternatively, organic aerogels are made from organic precursors that produce strong networks of covalently bound polymers, usually with resorcinol-formaldehyde or melamine-formaldehyde. These are produced in alkaline aqueous solutions through condensation processes, where $NaHCO_3$ and NaOH often serve as catalysts. The mechanical strength and flexibility of these materials are higher than those of their inorganic counterparts.
- **Carbon Aerogels:** By pyrolyzing organic aerogels at temperatures higher than 500°C, carbon aerogels are formed, which transform them into porous, electrically conductive carbon structures. The

porosity and surface area of these aerogels are highly dependent on the temperature at which they are pyrolyzed; the maximum surface area is typically observed at 600°C. The formation of closed cavities results from a significant decrease in pore volume at temperatures exceeding 2100°C. There are various physical forms of these aerogels, including films, granules, powders, and monoliths, as shown in Fig. 5.

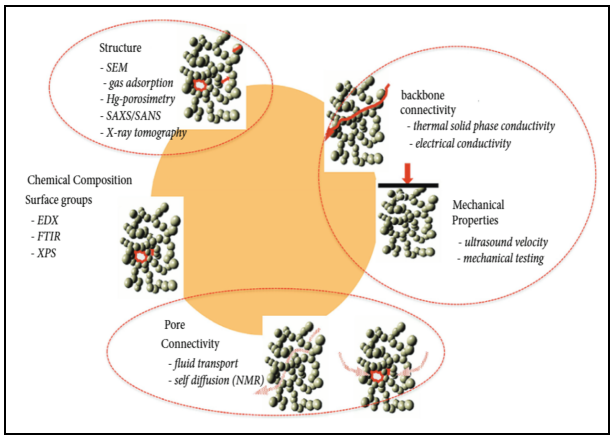


Fig. 5: Features of carbon-based aerogels (Arenillas *et al.* 2019)

- **Hybrid Aerogels:** These are obtained through the sol-gel process, which employs both organic and inorganic constituents. This combination enhances properties such as elastic modulus, hydrophobicity, and resilience. For instance, silica aerogels undergo organic incorporation, which makes the material mechanically stable and less brittle. The unique architecture of hybrid aerogels has increased the scope of application of modern aerogel materials in contemporary technology. The sol-gel method typically utilizes organometallic compounds, such as metal alkoxides or inorganic metal salts, as precursors. A series of condensation and hydrolysis reactions transforms these precursors into a sol, which is a colloidal suspension. The colloidal particles further undergo amalgamation into a continuous phase, gel-like substance. When this phase is subjected to drying and heating, it becomes a solid, resulting in dense glass or ceramic material. The fundamental reactions are (1) condensation of water, (2) condensation of alcohol, and (3) hydrolysis. Given the incompatibility of water and alkoxides, solvents like alcohols are often used to ensure sufficient mixing and accelerate the hydrolysis reaction. A smoother reaction process is facilitated by the alcohol's ability to mix the cellulose and alkoxide precursors evenly with water. Figure 6 lists the various types of materials that can be created using sol-gel technology and outlines the general steps of the process.

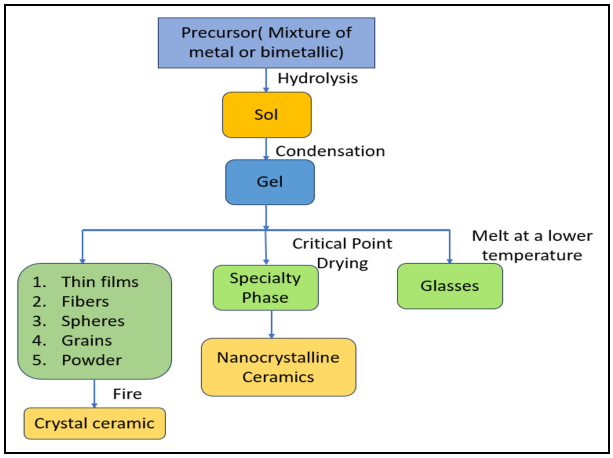


Fig. 6: An outline describing the several phases of the sol-gel process and the kinds of products that are produced (Kumar *et al.* 2015)

5. APPLICATIONS OF SOL-GEL

Many different materials have been synthesized and fixed onto various surfaces using the Sol-Gel technique. Light barriers, data storage, corrosion resistance, and conductivity enhancement are all facilitated by thin films with thicknesses ranging from nanometers to micrometers. They are ideal for solar cells that utilize materials such as TiO_2, SiO_2, and ZnO (Tan *et al.* 2014), whether pristine or modified with elements like Ag, due to their unique reflectance, absorption, and transmission properties. The Sol-Gel technique is also utilized in medicine and the environment, particularly for chemical sensing, where silica is employed in its pure state or covalently bound to significant and tiny molecules. Generally, synthesis techniques are optimized to control the composition, surface characteristics, and structure of metal oxide nanoparticles (MONPs) in order to enhance their corrosion resistance in harsh environments. In sol-gel or hydrothermal synthesis, doping with cerium or aluminum enhances passivation and encourages the formation of self-healing oxide layers. Corrosive agents are blocked by hydrophobic or chemically inert coatings made possible by surface functionalization with silanes or polymers. By fusing a stable shell with a functional core, core–shell structures—like FeO_4@SiO_2—provide extra protection. By enhancing crystallinity and decreasing defects, high-temperature annealing lessens the particles' reactivity to corrosive environments. Furthermore, uniformly dispersing MONPs in protective matrices such as silica derived from sol-gel reduces surface exposure and improves their stability over time. The Sol-Gel technique enables the modification of textile compounds by introducing various functional groups, enhancing properties such as water resistance, UV protection, tensile strength, abrasion resistance, thermal stability, and mosquito repellency. China exemplifies the use of Sol-Gel technology for energy conversion and renewable

energy, particularly in supercapacitors and fuel cells. This process facilitates the large-scale synthesis of nanomaterials for advanced energy applications and is essential to the development of safe, high-energy-density electrochemical devices. The application is illustrated in Fig. 7.

By serving as barriers against environmental stresses, coatings greatly increase the durability of plastics. In order to prevent polymer chain scission and surface cracking, UV-resistant coatings, for instance, use stabilizers like titanium dioxide or hindered amine light stabilizers (HALS), which absorb or scatter harmful UV rays. Water is repelled by hydrophobic coatings, which lowers moisture absorption and stops microbial growth, swelling, and hydrolysis brought on by moisture or rain. Furthermore, by protecting the plastic from sharp temperature changes, thermal barrier coatings or those containing ceramic fillers reduce brittleness or thermal expansion. In addition to prolonging the material's life, these coatings maintain its functionality, mechanical strength, and aesthetic appeal in challenging or outdoor settings.

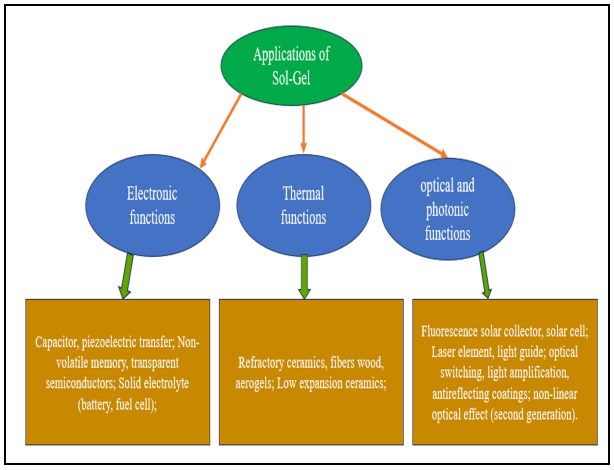


Fig. 7: Applications of sol-gel (Sakka *et al.* 2018)

5.1 Ceramics

"Ceramic" is derived from the Greek word "keramos," which means "potter's clay." Ceramics are generally crystalline, inorganic, and non-metallic materials. They are helpful in cutting-edge technologies due to their low density, excellent hardness, resilience to corrosion, and resistance to wear. A practical way for creating ceramics is the sol-gel method, which involves creating colloidal suspensions, or sols, that are subsequently dehydrated to produce gels. Afterwards, these gels undergo heat treatment, also known as calcination, to create thick ceramic materials. Initially, this technique was applied to glass-forming systems, particularly those made of alumina and silica. The sol-gel method has recently been extended to a variety of metal alkoxides, allowing for the production of fibers, composites, powders, and bulk monolithic components. Compared to conventional ceramic techniques, it offers a significantly more straightforward procedure in warm weather. Better control over the uniformity and purity of the end product is also made possible by the process. Despite its effectiveness, the sol-gel process requires careful handling due to its use of costly components, potentially hazardous solvents, and regulated preparation conditions. Alkoxides can be used to make ceramics cleaner and more stable by reducing the likelihood of ionic contamination when they are adequately cleaned. Since combining various sols can result in homogeneous microscopic compositions, the sol-gel process also facilitates the creation of multi-component materials. To keep homogeneity, it is necessary to control the various reaction rates of mixed alkoxides. Although the high liquid content can cause significant shrinkage, researchers have occasionally sintered gels straight into ceramics. Although the sol-gel process is excellent for making films, issues such as crazing, poor adhesion, and thermal mismatch between the substrate and the film need to be addressed. Notwithstanding these limitations, sol-gel processing remains a viable method for high-purity ceramic applications, particularly in advanced material engineering and electronics.

5.1.1 Advantage of Ceramics Made from Sol-gel

Compared to conventional metallic parts, the use of ceramic components in gas turbine engines offers several significant advantages. Among these areare the capacity to function at higher temperatures, which results in an increased thrust-to-weight ratio, decreased engine weight, and improved fuel efficiency. Ceramics also lessen the need for rare or strategic materials, which are frequently required for metallic alloys. At higher temperatures, ceramics retain their mechanical strength despite having a lower density than metals (Arenillas *et al.* 2019).

However, the majority of monolithic ceramics are intrinsically brittle and have low fracture toughness due to their atomic bonding structure, which renders them susceptible to abrupt failure, particularly under point contact stress. One of the main obstacles to their extensive use in engine applications has been their brittleness. Fiber-reinforced ceramic matrix composites (CMCs) have been created as a solution. Due to their significantly higher fracture toughness, these composites are a more reliable choice for high-performance ceramic engine parts, thereby reducing the likelihood of catastrophic failure.

5.1.2 Ceramic-carbon Composite Electrodes Made from Sol-gel

Ceramic-carbon electrodes (CCEs) possess several beneficial characteristics, including chemical modifiability, stiffness, porosity, and a reusable exterior surface. They are ideal for a wide range of

electrochemical applications because of these qualities. CCEs are more stable and long-lasting than conventional carbon paste electrodes. Additionally, CCEs exhibit greater chemical modification compatibility than organic and monolithic composite carbon electrodes, increasing their adaptability in sensor development and other cutting-edge technological applications.

5.1.3 Generated Ceramic Membrane from Sol-gel

The sol-gel modification of mesoporous alumina has proven to be an effective strategy for enhancing gas separation capabilities. The procedure generates a thin microporous top layer on the alumina surface, which enables active transport and molecular sieve-like separation. Gas separation efficiency and selectivity experience significant enhancements because of this process.

5.1.4 Ceramic-carbon Nanotube Nanocomposite Electrodes Derived from Sol-gel

The fabrication process of nanocomposite electrodes involves embedding multi-walled carbon nanotubes (MWNTs) into a silicate gel matrix through a sol-gel-derived process, resulting in ceramic-carbon nanotubes. The final result of this manufacturing procedure yields ceramic-carbon nanotube nanocomposite electrodes (CCNNEs). Researchers have thoroughly examined the electrochemical properties of these nanocomposites, which confirm their applicability in advanced electrochemical systems, including sensors, energy storage devices, and catalytic platforms.

5.2 Thin Film Coating

Silicon dioxide (SiO_2), titanium dioxide (TiO_2), magnesium fluoride (MgF_2), gold (Au), or aluminum (Al) are examples of ultrathin layers of materials that are deposited on the surface of various substrates, including metals, glass, ceramics, or crystals. These coatings change the underlying material's optical and electrical characteristics. Reflection, refraction, conductivity, and anti-reflection behavior can all be controlled by their thickness and composition. In contemporary technology, thin-film coatings are essential, particularly in solar panels, cell phones, flat-screen displays, optical lenses, and LEDs (Brinker *et al.* 1991). The intended performance and interaction with particular light wavelengths are taken into consideration while designing the coatings. To achieve the desired optical effects, several factors must be precisely calibrated, including the material chosen, the thickness of each layer, and the number of layers. The thickness of these coatings varies from a few nanometers to several micrometers (Salvaggio *et al.* 2016). They offer notable improvements to the substrate's mechanical, thermal, and optical properties despite their small size. They aid in the production of lightweight, high-performance materials when applied to plastic substrates, often replacing more costly and bulky substitutes like glass. However, when subjected to adverse environmental factors, such as UV rays, rain, or extreme temperatures, uncoated plastics may deteriorate (Tsvetkov *et al.* 2020).

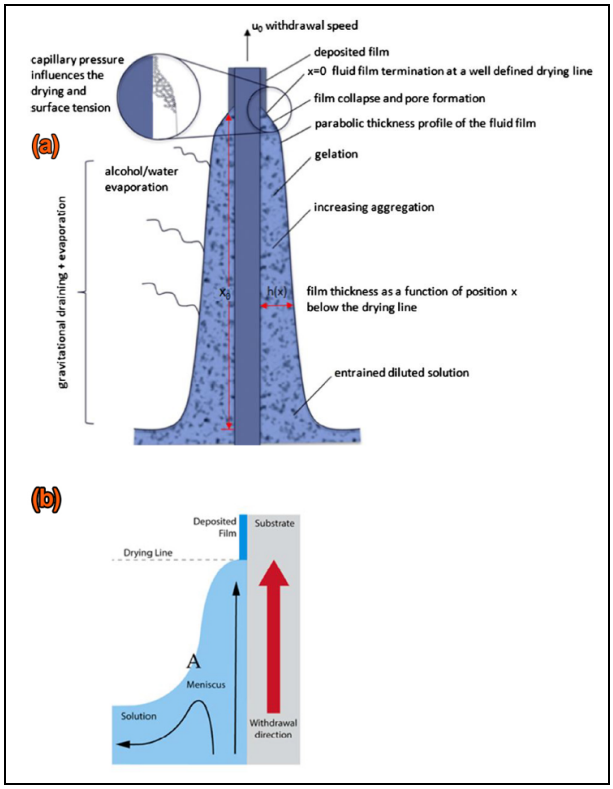


Fig. 8: (a) Process diagram for steady-state dip coating (Salvaggio *et al.* 2016) and (b) directions for streamlining the dip coating process (Wai *et al.* 2021)

Each of the various types of optical coatings serves a distinct purpose. Antireflection coatings minimize undesired reflection and glare, often at the interface between materials such as air and glass. Contrarily, high-reflection coatings are applied to mirrors to increase surface reflectivity. Beam splitters or partial reflection coatings separate light into transmitted and reflected areas at predetermined ratios. Polarized light is managed by polarization coatings, which allow one direction of light to pass through while reflecting the other. Filter coatings can be designed to transmit specific light ranges while reflecting others, depending on the wavelength (Salvaggio *et al.* 2016).

Dip coating is one of the most straightforward and most efficient sol-gel methods for applying thin films. This procedure involves dipping a substrate into a chemical solution and then carefully removing it at a predetermined pace. Viscosity and capillary forces cause the liquid to stick to the surface, forming the coating (Takahashi *et al.* 2006b). Following withdrawal, either forced or spontaneous, the coating solidifies through evaporation, as,as shown in Fig.Fig. 8. The ultimate

thickness and structure of the film can be precisely adjusted by controlling the temperature, vapor pressure, and withdrawal speed. Dip coating is suitable for both small-scale production and research, as it is relatively inexpensive and requires only basic equipment. By providing greater control over the film's microstructure, it offers a notable benefit over traditional techniques such as sputtering or chemical vapor deposition (CVD) (Chávez and Boccaccini, 2012; Corni *et al.* 2008).

Spin coating is another technique for applying thin films. This method involves depositing a liquid solution over the middle of a substrate, which is subsequently rapidly spun. The liquid is uniformly distributed across the surface by the centrifugal force. The excess fluid is thrown away, and evaporation causes the film thickness to decrease. Deposition, spin-up, spin-off, and evaporation are the steps that make up spin coating. Evaporation takes precedence over the others, particularly in sol-gel applications. As the solution dries, its viscosity rises, slowing the spin-off rate. Film thickness and homogeneity can be precisely controlled by varying the spinning duration and speed as well as the solution's characteristics (Tawa *et al.* 2019).

The sol-gel process's development from its original application in the manufacturing of glass to its use in sophisticated ceramics has significantly increased its adaptability and influence in a wide range of industries. The sol-gel process was initially created to create high-purity silica glass at low temperatures, but it has since expanded to include a variety of metal alkoxides and precursors, allowing for the creation of complex ceramic oxides with adjustable characteristics like TiO_2, ZrO_2, and Al_2O_3. This change made it possible to precisely control the morphology, porosity, and particle size—elements crucial for contemporary applications.

Consequently, sol-gel-derived ceramics are now widely used in energy storage devices, drug delivery systems, protective coatings, biomedical implants, and photocatalysts. For instance, sol-gel-coated electrodes increase battery stability and life, and bioactive glass-ceramics produced using sol-gel techniques exhibit superior bone bonding and resorption properties. Its versatility has also sparked advancements in thin films, aerogels, nanocomposites, and hybrid materials. As a result, the transition from glass to ceramics has made sol-gel processing a fundamental method for creating materials with multiple uses in both the scientific and industrial fields.

Table 2. Several materials for electronic and related applications that are produced from sol-gel (with typical sources of information)

Material	Deposition Method(s)	Key Properties	Applications
SiO_2 (Silicon Dioxide)	Dip coating, Spin coating	High transparency, chemical inertness, thermal stability, and antireflective behavior	Optical coatings, MOSFETs, and solar panels
TiO_2 (Titanium Dioxide)	Dip coating, Spin coating	Photocatalytic activity, UV absorption, hydrophilicity, and antibacterial	Solar cells, self-cleaning surfaces, biomedical coatings
ZnO (Zinc Oxide)	Dip coating, Spin coating	Semiconducting, photocatalytic, optoelectronic, UV-blocking	Photodetectors, dye-sensitized solar cells
MgF_2 (Magnesium Fluoride)	Dip coating	Anti-reflective, low refractive index, high optical clarity	Optical lenses and filters
$BaTiO_3$, $SrTiO_3$	Spin coating	Ferroelectric, high dielectric constant	Capacitors, memory devices, transducers
PLZT, PZT (Ferroelectric)	Spin coating	Ferroelectricity, piezoelectricity, and optical modulation	JFETs, actuators, optical switches
WO_3 (Tungsten Oxide)	Dip coating, Spin coating	Electrochromic behavior, switching capability	Smart windows, displays
$LiNbO_3$ (Lithium Niobate)	Sol-gel coating	Optoelectronic, piezoelectric, nonlinear optical properties	Waveguides, modulators, sensors
ITO (Indium Tin Oxide)	Dip coating	Transparent conductivity, infrared reflection	Touch screens, photovoltaics, display technology
V_2O_5, VO_2	Dip coating	Thermochromi, electrochromic, antistatic, semiconductor-metal transition	Energy-efficient windows, films
Hybrid Silica–Organic Coating	Electrophoretic deposition	Enhanced mechanical flexibility, hydrophobicity, and thermal resistance	Wear-resistant and protective coatings
Carbon Nanotube–Silica Hybrid	Sol-gel embedding	Electrical conductivity, mechanical reinforcement, and thermal stability	Electrochemical sensors, fuel cells
Al_2O_3 (Alumina)	Hot water treated sol-gel film	High hardness, wear resistance, and optical clarity (in flower-like films)	Antireflective coatings, corrosion-resistant layers
$CoCuMnO_x$ (Cobalt-Copper-Manganese Oxide)	Dip coating	High solar absorptivity (0.906), low emissivity (0.116), good adhesion	Solar selective coatings for thermal applications
Bioglass 45S5	Sol-gel synthesis	Bioactivity, bone bonding capability, controlled degradation	Bone tissue engineering, dental implants

Dip coating is a popular and affordable technique for applying consistent thin films to cylindrical or flat surfaces. By varying the withdrawal speed, solution viscosity, and number of dips, it provides exact control over film thickness. This method is perfect for making ceramic, optical, and biomedical coatings because it works very well with sol-gel materials. It is appropriate for both small- and large-scale applications, and it permits good surface coverage as well. Dip coating, however, is less successful on intricate or irregular geometries, frequently leading to uneven coatings or edge buildup. Defects related to drying, like peeling or cracking, can happen, particularly in thick films. Achieving coatings free of flaws depends on the sol's quality and stability. Overall, dip coating is still a flexible and successful method for functional surface engineering.

5.2.1 Creation and Alteration of Coatings Derived from Sol-gel

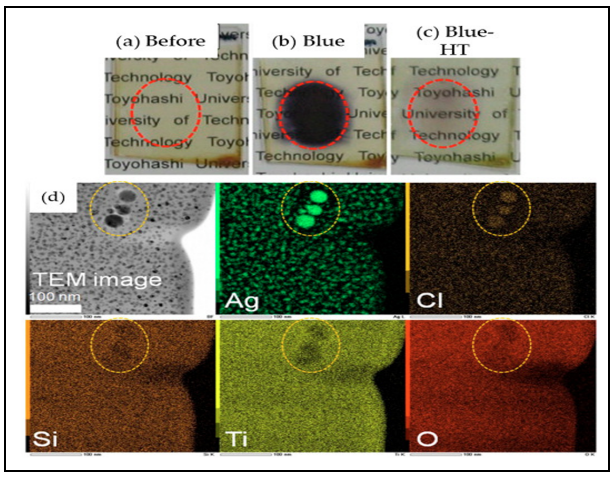


Fig. 9: (Ag15Cl:40GPSiO$_{3/2}$-60TiO$_2$ (GP: Glycidoxypropyl, mol%) film photographed before and after blue laser irradiation and heat treatment (a–c). (d) Following blue laser irradiation, a cross-sectional TEM picture and matching EDX mapping of a 30Ag15Cl:40GPSiO$_{3/2}$-60TiO$_2$ layer. In each part, the circles represent the same area (Kawamura *et al.* 2018)

5.2.1.1 Sol-gel Coatings Exposed to Photo-irradiation

An efficient method for local densification before selective etching is the micropatterning of sol-gel-generated films using laser photoirradiation. To enable localized photoreaction, researchers first altered metal alkoxides with β-diketones (Kawamura *et al.* 2011), which absorb UV light and show π–π* transitions. The exposed regions were selectively leached using ethanol or aqueous nitric acid following UV irradiation through a photomask, and the films were heat-treated to eliminate any remaining organics. Using $SnCl_2$ and acetylacetone as precursors, this method was used to pattern conductive tin oxide films. Following UV exposure, the movie underwent heat treatment at 500°C and was leached with either NaOH or NH_4OH. Between 3 and 50 µm in width and 0.1 µm in thickness, the generated patterns exhibited a resistance of approximately $1 \times 10^2 \Omega \cdot cm$.

For holographic applications, researchers employed blue laser irradiation on AgBr-doped organo-silsesquioxane–TiO_2 films. AgBr was transformed into Ag nanoparticles by the laser, which enhanced the film's visible absorption and enabled two-beam interference exposure. Later, it was shown in Fig. 9Fig. 9 that reversible recording was possible by irradiating AgCl to form Ag nanoparticles and then heat-treating it back to AgCl. By stabilizing Cl ions and increasing film density, TiO_2 prevented leaching and enabled steady film regeneration without morphological alteration. This innovation created new avenues for utilizing films made from sol-gel for rewritable optical storage.

5.2.1.2 Sol-gel Film Electrophoretic Patterning

Electrophoretic deposition (EPD) is a flexible method that uses an electric field to create thin or thick layers on conductive surfaces. Due to its rapid rate of deposition, thick films can be formed, and the voltage, deposition duration, and suspension concentration can all be accurately adjusted to control the film thickness. For the development of high-quality films, a stable suspension with a consistent particle distribution is essential. For compatibility and consistency in the final film, sol-gel generated particles are used in electrophoretic sol-gel deposition. For micropatterning applications, where thick transparent films are used as micro-optical components and wavelength guides, this method is beneficial. This study demonstrates EPD's potential for creating accurate, patterned optical coatings. Some researchers showed how to micropattern benzyl-silsesquioxane ($BnSiO_{3/2}$) films on indium tin oxide glass.

Before electrophoretically depositing the $BnSiO_{3/2}$, they created hydrophobic–hydrophilic patterns using the sol–gel technique. When $BnSiO_{3/2}$ is heated above the glass transition temperature in Fig. 10, it migrates from hydrophobic to hydrophilic regions. The development of convex-shaped micropatterns was validated by three-dimensional surface and cross-sectional profiles.

This work demonstrates the promise of EPD in creating accurate, patterned optical coatings. Scalability and reproducibility are two areas where electrophoretic deposition (EPD) is limited. It can be difficult to maintain a consistent electric field on larger substrates, which results in uneven film thickness. Particle movement and deposition can also be impacted by variations in substrate geometry, conductivity, or surface roughness, which lowers pattern repeatability and precision. Furthermore, it is challenging to standardize the process for industrial applications due to the complexity of controlling the deposition parameters (voltage, time, and particle concentration) across different scales.

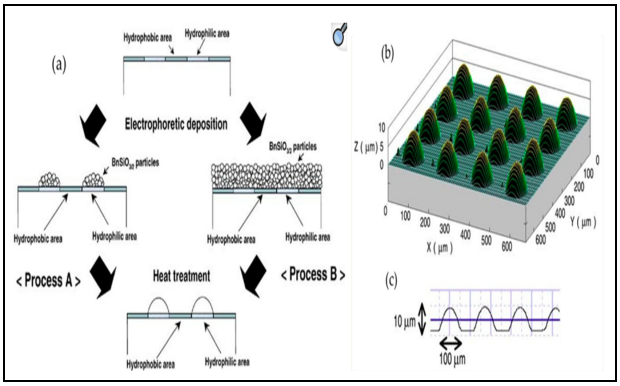


Fig. 10: (a) Diagram for creating $BnSiO_3/2$ micropatterns employing a hydrophobic–hydrophilic surface pattern and electrophoretic sol–gel deposition (b) three-dimensional surface profile and (c) cross-sectional profile of convex-shaped $BnSiO_3/2$ micropatterns made by electrophoretic sol-gel deposition with a hydrophobic–hydrophilic surface pattern and 30 minutes of heat treatment at 200 °C (Takahashi *et al.* 2007)

5.2.1.3 HWT of Al_2O_3-based Sol-gel Derived Coatings Produces Layered-double Hydroxide Films

Recent work has explored the application of hot water treatment (HWT) in the synthesis of layered double hydroxides (LDHs). (Yamaguchi *et al.* 2007) first described the preparation of platelet-shaped Zn-Al LDH by HWT of pre-treated sol-gel-derived Al_2O_3-ZnO thin films at 400 °C for 30 minutes. The thin films were prepared from aluminum tri-sec-butoxide ($Al(O\text{-}sec\text{-}Bu)_3$) and zinc acetate dihydrate ($Zn(OAc)_2{\cdot}2H_2O$). Notably, the Zn-Al LDH structures formed in situ on glass substrates after HWT, which is a straightforward and efficient method for immobilizing Zn-Al LDH. Nevertheless, the rapid precipitation of large LDH crystals caused visible light scattering, resulting in the films becoming opaque after just one minute of immersion. To answer this, a follow-up work aimed at controlling the precipitation process to produce transparent LDH films more appropriate for optical study of anion-intercalated LDHs. The Zn/Al ratio was found to affect the surface structure of the films, with an equimolar Zn/Al ratio generating the highest density of well-ordered hexagonal structures. Lowering the HWT temperature from 100 °C to 65 °C resulted in smaller crystal precipitates. Interestingly, even after 3 hours of HWT, as illustrated in Fig. 11, the films retained a transmittance greater than 50%, indicating that transparency might be maintained under ideal treatment conditions. The researcher was able to intercalate Eosin Y dye into Zn-Al LDH films with the aid of HWT in distilled water containing 5 mM Eosin Y at 100°C for 30 minutes. Intercalation of the dye in the LDH layers significantly enhanced thermal stability of the films compared to the surface-only Eosin Y adsorbed films. This was due to the protective role of the LDH matrix, which shielded the dye from oxidation by ambient oxygen. In addition, the Eosin Y-intercalated Zn-Al LDH films also showed promise for dye-sensitized solar cells applications, as they successfully operated under visible light illumination.

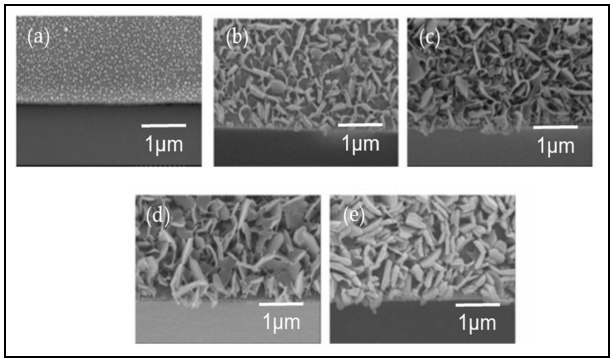


Fig. 11: Tilted cross-sectional FESEM pictures (20°) of Al_2O_3-ZnO film (Zn/Al = 1) coated on soda lime glass substrates following (a) heat treatment at 400°C (without HWT), followed by HWT at 65°C for (b) 1 min, (c) 15 minutes, (d) 3 hours, and (e) 4 hours (Yamaguchi *et al.* 2006)

5.3 Formation of Porous Ceramic Films

The sol-gel process is also widely used for fabricating three-dimensional porous ceramic structures. Microporous and mesoporous ceramics, in particular, have garnered considerable interest due to their efficacy in adsorption systems and drug delivery applications, resulting in significant improvements in this class of materials, by altering the sol-gel chemistry, pore size, and network density can be customized to regulate the release of several medications in a sol-gel matrix. Staggered release can be achieved through sequential loading or layering, and drug-property-based selective diffusion can be achieved through surface functionalization. Stability and control over release are further improved by adding polymers or encapsulating medications in carriers. (Jittiarporn *et al.* 2017). Sol-gel-based techniques have effectively manufactured porous ceramics, such as as zirconium oxide (ZrO_2) (Ismail, 2016a), silicon carbide (SiC), aluminum oxide (Al_2O_3), and wrinkled silica (SiO_2), as well as core-shell composite materials. Zirconium oxide stands out due to its exceptional chemical stability, making it an ideal material for use in sensors and as an adsorbent. Using Pluronic P123 as a template during the sol-gel process allows tuning the crystal structure of ZrO_2 to be amorphous, tetragonal, or monoclinic by adjusting the annealing temperature. Adjusting the film deposition settings might also result in hierarchical pore architectures. Changing the spin-coating speed and the number of layers deposited allowed them to regulate film thickness and porosity at both the meso- and macroporous levels. The ZrO_2 films, featuring a dual microstructure of worm-like mesopores and macropores, exhibited good optical transparency in the visible spectrum, underscoring their potential in sensor and catalytic applications. Hollow ceramic spheres have also sparked widespread interest, particularly for

medicine administration and cosmetic uses. Katagiri et al. developed a one-step process to synthesize hollow nanospheres of titanium dioxide (TiO_2) and strontium (Li *et al.* 2021) titanate ($SrTiO_3$). Hydrothermal treatment resulted in both shell crystallization and core disintegration, yielding hollow nanospheres with a diameter of 200 nm. The sol-gel method was used to create monodisperse silica gel particles, which were then coated layer-by-layer (LbL) with titanium (IV) bis (ammonium lactato) dihydroxide (TALH), a weak polyelectrolyte, as well as poly (sodium p-styrene sulfonate) (PSS) and poly (diallyldimethylammonium chloride) (PDDA). This LbL assembly, driven by electrostatic interactions, resulted in a uniform TALH coating on the silica nanospheres.

5.4 Templates for Generating the Plasmonic Effect

Photocatalytic materials like ZnO and TiO_2 are limited by their capacity to absorb light mainly in the ultraviolet (UV) region, which accounts for just 3-5% of the solar spectrum. This limited light absorption considerably reduces their efficiency in solar-powered applications. To overcome this constraint, researchers investigated the incorporation of metal nanoparticles (NPs) onto oxide films to increase their absorption into the visible light spectrum. This improvement is made possible by the surface plasmon resonance (SPR) phenomenon, which happens when metal NPs interact with light, increasing the local electromagnetic field and hence the material's overall light absorption capabilities. The material's surface area is an essential aspect in optimizing the SPR effect and overall photocatalytic effectiveness. Mesoporous templates—structures with well-defined pore systems—are frequently used to support and arrange the deposition of metal nanoparticles. The sol-gel method has developed as a highly effective technology for producing mesoporous structures, allowing for fine control over porosity, surface shape, and chemical composition. Sol-gel-derived SiO_2-TiO_2 mesoporous template to deposit gold nanoparticles (Au NPs) and gold nanorods (Au NRs) in tubular mesochannels, resulting in Au/SiO_2-TiO_2 composite materials. Au-NR-decorated composites outperformed Au-NP-based composites and conventional TiO_2 (P25) films in terms of photocatalytic activity for methylene blue degradation. The superior performance was attributed to two main factors: first, the high surface area of the sol-gel template facilitated effective formation of Schottky barriers between Au and TiO_2, improving charge separation. Second, the tubular mesochannels provided an ideal environment for the alignment and incorporation of Au nanorods, which absorbed light in the visible and near-infrared ranges, extending the material's photoresponse. Other studies have expanded on this concept by investigating the construction of core-shell nanostructures to improve photocatalytic and photoelectrochemical efficiency. Nbelayim et al. produced Ag@TiO_2 core-shell nanoparticles using a sol-gel technique for dye-sensitized solar cells (DSSCs). The silver core boosted light absorption through significant plasmonic effects, while the TiO_2 shell stabilized the semiconductor interface. This combination enhanced both optical characteristics and the Fermi level of TiO_2, resulting in more efficient charge injection and increased device performance (Wu *et al.* 2020).

These investigations demonstrate the versatility of the sol-gel method in developing functional mesoporous and nanostructured materials. The sol-gel technique, which allows for the precise construction of templates for metal NP inclusion, continues to play an important role in the advancement of photocatalytic and energy-conversion technologies through the fabrication of highly efficient plasmonic composites.

5.5 Waste Removal Using Extraction Technology

Rapid industrial development to meet consumer needs has resulted in substantial waste generation, which has severely polluted essential water sources. Among the contaminants, bisphenol A (BPA) is a serious problem. BPA, which is commonly found in plastics used for food containers, drinks, and medical equipment, is renowned for its propensity to mimic natural hormones, altering endocrine function and endangering human health. To address this issue, (Kalogiouri *et al.* 2020). Created molecularly imprinted polymers with a sol-gel matrix for targeted BPA extraction from water. These polymers demonstrated exceptional selectivity and absorption capacity, as well as significant retention of extraction efficiency after 10 reuse cycles, demonstrating both performance and sustainability, as shown in Fig. 12.

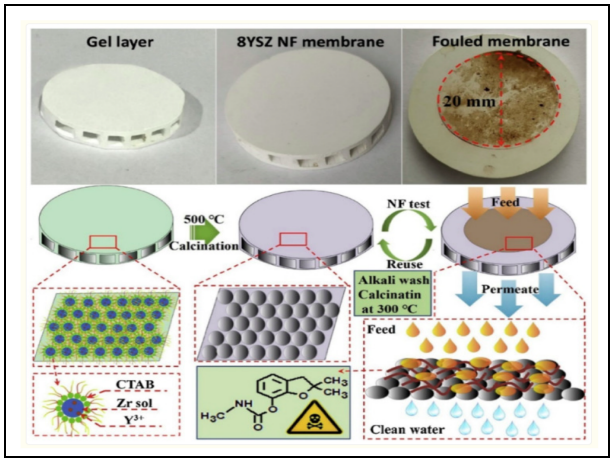


Fig. 12: The creation of 8-mol% yttria-stabilized ZrO_2 nanofiltration membranes using a reverse micelles-mediated sol-gel method and carbofuran removal (Qin *et al.* 2020)

In addition to organic pollutants such as BPA, heavy metals provide a significant difficulty in

wastewater management due to their toxicity and long-term health consequences. The sol-gel process has emerged as a powerful way for producing sophisticated materials that can effectively remove these impurities. Researchers employed sol-gel techniques to produce electrospun Fe_3O_4 fibers, sodium-modified mesoporous TiO_2, ultrafine alumina fibers, and yttria-stabilized ZrO_2 membranes. They successfully created nanofiltration membranes from size-controlled ZrO_2 nanoparticles using a reverse micelle-assisted sol-gel technique. These membranes were explicitly designed for pesticide removal, and they effectively removed carbofuran from water with an impressive 89% efficiency. Incorporating yttria into the ZrO_2 matrix reduced the phase transition from tetragonal to monoclinic, improved structural stability, surface area, and filtration performance (Lee *et al.* 2018).

When creating nanofiltration membranes, the reverse micelle-assisted sol-gel technique has a number of significant advantages over conventional sol-gel techniques, especially with regard to performance, uniformity, and control over the nanostructure. This is a succinct explanation:

- Reverse micelles function as nanoreactors, containing sol-gel reactions within droplets. This enables highly controlled particle size (usually 1–10 nm) and narrow distribution, which is perfect for forming homogeneous membrane pores.
- **Increased Porosity and Pore Size Tunability:** The method allows for fine-tuning of pore sizes and distribution, which is essential for selective separation in nanofiltration applications, by varying the surfactant and water-to-surfactant ratios.
- **Improved Homogeneity:** The micellar environment makes sure that precursors are mixed more effectively at the molecular level, which produces materials that are extremely homogeneous and have fewer agglomerates or defects.
- **Best Membrane Performance:** Because of their uniform nanoporous structure and defect-free morphology, the resultant membranes frequently show higher flux, better selectivity, and greater mechanical/chemical stability.
- **Low-temperature Processing:** This technique maintains the benefit of low-temperature synthesis, which makes it suitable for heat-sensitive substrates or functional additives, just like traditional sol-gel.

This research highlights the importance of sol-gel-generated materials in creating enhanced waste remediation solutions. These materials make a substantial contribution to developing sustainable water management solutions and supporting broader environmental goals due to their high selectivity, reusable nature, and efficiency.

5.6 Sol-gel Templating to Form Nanocomposites

Multiferroic nanocomposites, which exhibit both ferromagnetic and ferroelectric properties, have garnered significant attention due to their potential applications in advanced technologies such as data storage, sensors, and microwave transducers. Historically, the manufacturing of these composites necessitated expensive and difficult physical alignment processes. However, recent studies' interests have shifted toward more cost-efficient and scalable technologies, among which liquid phase methods, such as the sol-gel technique, are advantageous because they allow for better control of composition and structure at the nanoscale. The sol-gel method has presented tremendous potential in the fabrication of film-type and heterostructured multiferroic composites.

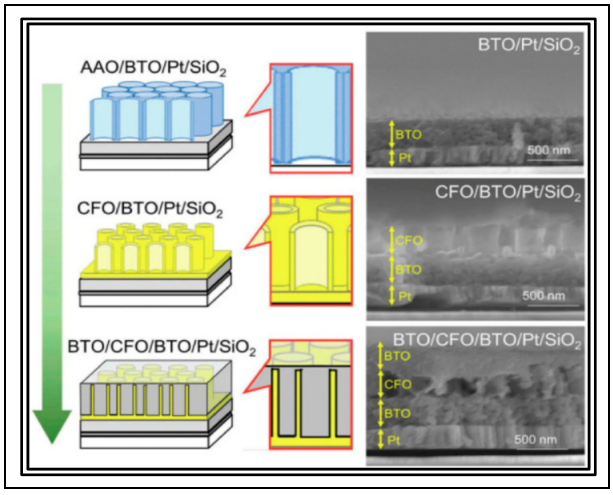


Fig. 13: A schematic of the fabrication method for the multiferroic nanocomposite, BTO/CFO/BTO, on a Pt-coated SiO_2 substrate. The relevant SEM images are given from top to bottom: (AAO-placed) BTO compact layer on a Pt-coated SiO_2 substrate; CFO nanotube arrays on BTO/Pt/SiO_2; and BTO-coated CFO nanotube arrays on BTO/Pt/SiO (Kawamura *et al.* 2011)

The most known example is the fabrication of $BaTiO_3$ (BTO)-$CoFe_2O_4$ (CFO) nanocomposite. BTO, a classical ferroelectric, was used to form the bottom layer by sol-gel spin-coating on a platinum-coated SiO_2 wafer. The CFO, being a ferrimagnetic material, was subsequently deposited using a sol-gel spin-coating process in a nanoporous anodic alumina oxide (AAO) template which produced a well-aligned structure. The resulting structure, after chemical etching of the AAO template with NaOH, was a BTO layer with CFO nanotubes (Periyasamy *et al.* 2020).

To complete the composite, another BTO layer was spin-coated on top, resulting in a vertically aligned structure with ferrimagnetic CFO nanopillars embedded in a ferroelectric BTO matrix, as shown in Fig. 13. This carefully constructed architecture allowed for high coupling between the electric and magnetic phases, which is essential for multiferroic activity. The composite exhibited distinct dielectric hysteresis loops, and its ferroelectric properties were controllable by an external magnetic field, demonstrating the successful integration of multiferroic functionality. This method not only reflects the adaptability and effectiveness of sol-gel techniques in synthesizing complex nanocomposite materials, but it also opens up new avenues for producing cost-effective, multifunctional devices for future technologies.

5.6.1 Metal Oxide Nanoparticles via Sol–gel Method

Metal oxide nanoparticles (MONPs) exhibit unique physical, chemical, optical, and electronic properties that differ significantly from those of their bulk counterparts. Quantum confinement effects and the higher density of reactive surface atoms at the nanoscale primarily bring about these distinctions. The functionality and application value of MONPs are predominantly related to such crucial features as surface area, particle size and shape, crystallinity, stability, corrosion resistance, electrical conductivity, and photocatalytic activity. For effective commercialization, rigorous control over synthesis parameters becomes essential to achieve reproducibility and uniformity.

The methods of synthesis for MONPs are usually divided into two categories:

(i) Physical methods, which take a top-down strategy, consist of disintegrating bulk materials into particle sizes in nanometers. The techniques in this category are ball milling, sputtering, laser ablation, electrospraying, and electron beam evaporation.

(ii) Chemical methods, following a bottom-up synthesis concept, involve the creation of nanoparticles through the controlled nucleation and growth of atoms or molecules. Typical chemical synthesis methods are sol-gel processing, the polyol process, hydrothermal and solvothermal synthesis, co-precipitation, microemulsion methods, and chemical vapor deposition (CVD).

Physical methods are usually simpler and easier to scale, yet chemical routes provide better control over particle morphology, composition, and size distribution (Matsuda *et al.* 2000), (Matsuda *et al.* 2005a).

5.6.2 Sol-gel Technique for Synthesizing TiO_2 Nanoparticles

The sol-gel process is a straightforward, low-cost, and effective method for preparing titanium dioxide (TiO_2) nanoparticles (NPs) (Tsvetkov *et al.* 2020), (Takahashi *et al.* 2006). Various research groups have used sol-gel technology to prepare TiO_2 nanostructures with tailored properties. In a standard sol-gel reaction, TTIP reacts with ethanol under hydrolysis to produce TiO_2 NPs, and nitric acid is used as a catalyst to facilitate the hydrolysis and condensation processes. Research has proved that TiO_2 nanoparticles synthesized through the sol-gel process contain greater crystallinity and reduced crystallite sizes in comparison with the hydrothermally synthesized TiO_2 NPs under comparable conditions. The particle size of the TiO_2 NPs was determined to be around 9 nm (Matsuda *et al.* 2006; Matsuda *et al.* 2005b).

5.6.3 Mechanical Properties

Ceramics, and inorganic materials in general, are recognized for their excellent mechanical properties, like hardness, tensile strength, elasticity, etc. These traits arise from the fact that their chemical structures are predominantly composed of strong ionic and covalent bonds, far stronger than metallic bonds. The same bonding characteristics that impart tremendous strength to these materials also limit the ability for plastic deformation, making these materials inherently brittle. This brittleness leads to "catastrophic fracture" where the cracks propagate very rapidly and there is no warning as the structure fails because it cannot dissipate stress. This limitation is typical of other sol-gel inorganic materials such as aerogels (Pirzada *et al.* 2012*)*. This high porosity, which can be as high as 98% in some aerogels, further limits mechanical strength. For example, a TMOS-derived, 95% porous silica aerogel has a flexural strength of only 0.02 MPa, a tiny percentage of that of dense silica.

Given these constraints, enhancing the mechanical performance of sol-gel materials is crucial for their use in bulk or coating applications. The mechanical properties of these materials are essentially determined by their internal network architectures, which can be customized using synthesis parameters such as the water-to-precursor molar ratio, pH, precursor type, and dopants. For example, increasing water content hastens hydrolysis reactions while decreasing material density due to dilution effects. Murtagh et al. found that increasing the water content of TEOS-based silica aerogels reduced their elastic, shear, and bulk moduli. The pH of the solution also plays an important role. While it has a direct impact on the rate of hydrolysis and condensation, it also impacts the gel's drying behavior and mechanical integrity. discovered that increasing the pH of a TEOS-based sol reduced shrinkage during drying from 15% to 1.5%, owing to the development of larger, stiffer silica particles at higher pH values. Reduced shrinkage results in fewer internal tensions and increased mechanical stability.The type of precursor employed is another critical consideration. For example, xerogels

made from TMOS tend to be stronger than those made from TEOS under similar conditions. However, despite their strength, typical silica-based materials are fragile. To address this, researchers have investigated flexible and hydrophobic aerogels made from alkylalkoxysilane precursors such as MTMS and MTES. These precursors add non-reactive alkyl groups to the network, lowering inter-chain cohesion while improving flexibility (Hawthorne *et al.* 2004).

Table 3. Improved mechanical properties by the sol-gel method

Material / Composite	Sol-Gel Strategy	Improved Mechanical Properties	Enhancement Mechanism
Silica Aerogels (SiO_2)	Adjusting pH & water-to-alkoxide ratio	Shrinkage reduced from 15% to 1.5%, improved stiffness and modulus	Larger silica particles at higher pH reduce internal stress
Silica–Chitosan Hybrid Xerogels	Organic-inorganic hybridization	Compressive strength increased from 10 MPa to 70 MPa	Hydrogen bonding and interpenetrating networks
MTMS-/MTES-based Silica Aerogels	Use of hydrophobic alkylalkoxysilanes	Increased elasticity and hydrophobicity	Flexible network formation with alkyl chains
45S5 Bioactive Glass (with TiO_2/MgO)	Dopant addition during sol-gel synthesis	Improved bending strength, hardness, and fracture resistance	Ionic substitution stiffens the silica network
TiO_2/SiO_2 Nanocomposites	Mixed oxide synthesis	Enhanced surface hardness and scratch resistance	Synergistic phase interaction and particle reinforcement
Mesoporous ZrO_2 Films	Template-assisted sol-gel with Pluronic P123	Maintains structural integrity under load, good adhesion	Hierarchical pore structure + ZrO_2 crystallinity
Sol-Gel Alumina Coatings on Steel	Thermal treatment of Al-based sol-gel precursors	Increased wear and corrosion resistance, higher hardness	Formation of dense crystalline coating
PVA–Silica Nanofibers	Electrospinning of sol-gel derived silica-polymer mix	Tough, flexible nanofibrous mats	Homogeneous silica dispersion in polymer matrix
Silica-Coated Textiles	Sol-gel coating with functional silanes	Increased abrasion resistance, tensile strength, and durability	Covalent bonding between sol-gel network and the fabric fibers
TiO_2–Carbon Nanotube Composites	Embedding CNTs into the TiO_2 matrix via sol-gel	Higher fracture toughness and mechanical strength	Crack bridging and load transfer by CNTs
SiO_2–PCL Hybrid Scaffolds	Biopolymer (polycaprolactone) integrated with silica	Improved compressive modulus and elasticity	Interpenetrating networks of organic-inorganic domains
$BaTiO_3$–$CoFe_2O_4$ Nanocomposites	Spin-coated heterojunctions using sol-gel	Mechanically stable multiferroic stacks	Template-assisted growth and nanotube alignment

The mechanical characteristics of silica networks become enhanced when they are combined with other elements. The network becomes more robust when Si or O atoms are replaced by ions such as Mg^{2+} or Ti^{4+} because these substitutions alter the bond lengths and coordination. The bending strength, fracture toughness, and hardness of 45S5 bioactive glass were enhanced through the addition of MgO or TiO_2. This is due to the dopants' higher ionic properties and shorter bond lengths. High-temperature treatments, such as sintering, are often ineffective in drug delivery systems (DDS) due to the risk of damaging delicate therapeutic ingredients (Jelle *et al.* 2015). To overcome this, hybrid organic-inorganic systems have gained popularity. These materials enhance flexibility and mechanical performance by combining sol-gel silica with biocompatible and biodegradable polymers, such as chitosan, collagen, gelatin, PCL, and PLLA. For example, a hybrid silica-chitosan xerogel with a notable improvement in compressive strength—from 10 MPa to 70 MPa—as well as increased flexibility (Toonder *et al.* 2002). This hybrid system also demonstrated controlled, prolonged medication release with adjustable release profiles dependent on chitosan content. Their later work with silica-chitosan membranes and coatings showed the promise of these hybrids in biomedical applications. These achievements underscore the significance of material engineering in overcoming the inherent brittleness of sol-gel produced ceramics and broadening their application in advanced technologies.

Methyltrimethoxysilane (MTMS) and methyltriethoxysilane (MTES), two hydrophobic and flexible precursors, have a major impact on the mechanical characteristics and microstructure of sol-gel materials. Because of the disruption of the dense silica network caused by their organic methyl groups, the cross-linking density is reduced, increasing porosity and flexibility. As a result, materials have better resistance to cracking, increased mechanical compliance, and decreased brittleness. Non-polar methyl groups also add hydrophobicity, which improves water resistance and stability over time in humid environments. However, because there is less inorganic content, hardness or stiffness may be slightly compromised when compared to pure silica systems.

5.7 Sol-gel in Biomedical Applications

The sol-gel technique is one of the most versatile and commonly used methods for preparing advanced materials, offering excellent control over their structure, composition, and properties, and is thus especially important in biomedical fields. It is a process in which a colloidal solution (sol) is converted into a three-dimensional gel network through the action of hydrolysis and condensation reactions at relatively low temperatures. These soft processing conditions facilitate the inclusion of sensitive biomolecules, drugs, and polymers without losing their bioactivity.

The sol-gel method enables the synthesis of a diverse range of material structures, spanning nanoparticles to thin films, coatings, monoliths, and porous scaffolds. Materials obtained from this procedure are typically characterized by a high surface area and tunable porosity, which enhances their interaction with biological environments—a valuable property for applications such as drug delivery, bone tissue engineering, and implant surface modification. Additionally, the inherent versatility of the sol-gel process enables the creation of organic-inorganic hybrid materials that combine the mechanical strength of inorganic phases with the functional flexibility of organic polymers (Xu *et al.* 2018).

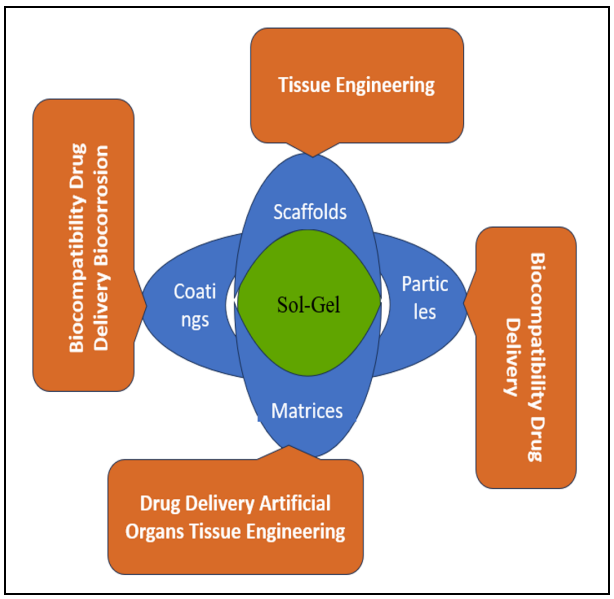


Fig. 14: Various applications of sol-gel materials in the biomedical industry (Aurobind *et al.* 2006)

Figure 14 illustrates that Sol-gel chemistry has been widely employed to produce silica-based materials, phosphate glasses, and metal oxide systems (e.g., TiO_2, ZnO, and Fe_3O_4), each with distinct biological properties. Silica-based sol–gel materials are recommended for their biocompatibility, structural tunability, and ability to host different medicines and bioactive compounds. They are commonly utilized in drug delivery systems (DDS), where mesoporous silica nanoparticles (MSNs) allow for controlled release and targeted distribution. Similarly, temporary implants and injectable cements use phosphate-based sol-gel materials, which are biodegradable and resorbable. Sol-gel synthesis produces metal oxides, such as TiO_2 and ZnO, which are utilized in phototherapy, imaging, antibacterial coatings, and biosensing due to their optical, catalytic, and electrical properties (Wu *et al.* 2017). Polymer-assisted sol-gel methods enhance the potential of these materials by increasing their textural and chemical homogeneity, thereby allowing for greater bioactivity and integration with host tissues, as shown in Fig. 15.

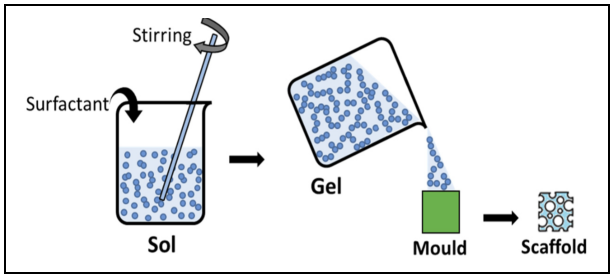


Fig 15: Creation of scaffolds using the sol-gel synthesis method for tissue engineering applications (Baino *et al.* 2019)

The following are the most crucial prerequisites for creating scaffolds for bone tissue engineering that can stimulate tissue growth:

- **Biodegradability:** The scaffold must eventually be modified by osteoblast activity and deteriorate at the same pace as the new tissue grows at the implant site.
- **Osteogenesis:** In addition to preventing fibrous tissue from encapsulating, the scaffold should be able to form an osteoconduction link with the host bone. For the scaffold to produce new bone, cells that can undergo further differentiation must be drawn to the implantation site.
- **Biocompatibility:** The scaffold must facilitate attachment, proliferation, and cell differentiation in vivo, be biocompatible and bioactive, and not elicit any adverse inflammatory reactions or toxic byproducts.
- **Fabrication:** The scaffold should be able to be readily formed into a variety of irregular shapes that can be used to replicate a patient's unique bone abnormalities.
- **Porous Microstructure:** A scaffold with interconnected pores and a high porosity (>80%) is suitable. A minimum diameter of 100 µm is required for a hierarchical porosity to facilitate neovascularization, tissue ingrowth, and cell penetration.
- **Mechanical Strength:** Scaffolds should possess the same mechanical strength as the bone tissue being replaced. During tissue regeneration and breakdown, the scaffold intended for regeneration should offer support.
- **Commercialization:** The manufactured scaffold should be capable of being commercialized. The worldwide standards and guidelines for the usage of these in biomedical equipment should be followed when performing sterilization (Ismail, 2016b).

Sol-gel-derived materials play essential roles in tissue engineering, orthopaedics, dentistry, and diagnostics. Porous bioactive glass scaffolds promote cell adhesion, proliferation, and differentiation, making them ideal for bone and soft tissue regeneration. In drug delivery, sol–gel systems enable prolonged and stimuli-responsive release, thereby enhancing therapeutic efficacy and reducing side effects. Additionally, functionalized sol-gel nanoparticles are utilized in imaging and biosensing applications to improve diagnostic sensitivity and specificity. Sol-gel coatings on medical implants enhance biocompatibility, reduce the risk of infection, and promote osseointegration. While there are still limitations, such as scalable production and cost-effective processing, the sol-gel method remains an essential enabling technology for next-generation biomedical devices. Its potential to effortlessly integrate with biological systems and assist the development of multifunctional, biocompatible materials keeps sol-gel chemistry at the forefront of materials science and biomedical innovation (De *et al.* 2014).

9. CONCLUSIONS

- The sol-gel process has evolved as a highly versatile and chemically accurate technique for synthesizing a diverse range of innovative materials, offering distinct benefits in terms of microstructural and functional properties.

- It enables the production of nanostructured ceramics, hybrid composites, and porous materials at mild conditions, allowing superior control over porosity, surface area, and mechanical strength as compared to traditional synthesis processes.

- Due to its low processing temperature, low waste, and use of green solvents like ethanol or water, the sol-gel method is environmentally friendly. When compared to traditional ceramic techniques, it uses less energy. Its environmental impact is negligible when eco-friendly precursors and appropriate solvent recycling are used.

- Despite the intrinsic brittleness of sol-gel-derived materials, significant progress has been achieved in improving mechanical integrity through:

    a) Optimising process parameters (for example, water-to-precursor ratio and pH)

    b) Elemental doping, such as Mg^{2+} and Ti^{4+}

    c) Using flexible and hydrophobic precursors (e.g., MTMS, MTES)

    d) Organic-inorganic hybridization with biocompatible polymers, including gelatin, collagen, and chitosan

- The method allows for multi-functionality, such as in:

    a) Biomedical applications include medication delivery systems and scaffolds for tissue engineering.

    b) Environmental remediation involves the selective removal of organic contaminants (e.g., BPA) and heavy metals.

    c) Optical and electronic device creation, including antireflective coatings, sensors, and multiferroic composites.

- Sol-gel systems' low-temperature processing capacity provides compatibility with delicate biological or medicinal agents, which is critical when designing bioactive and biodegradable materials.

- Future advances in sol-gel chemistry, such as templating techniques, nanocomposite integration, and 3D micro-structuring, are likely to stimulate additional innovation in sustainable material solutions.

- Overall, the sol–gel technique is a cornerstone technology in materials science, enabling the design and scalable manufacture of next-generation materials for high-performance and environmentally responsive applications.

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CONFLICTS OF INTEREST

The authors declare that there is no conflict of interest.

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