Review Article

Nanomaterial by Sol-Gel Method: Synthesis and Application

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The sol-gel process is a more chemical method (wet chemical method) for the synthesis of various nanostructures, especially metal oxide nanoparticles. In this method, the molecular precursor (usually metal alkoxide) is dissolved in water or alcohol and converted to gel by heating and stirring by hydrolysis/alkoxideysis. Since the gel obtained from the hydrolysis/alkoxideysis process is wet or damp, it should be dried using appropriate methods depending on the desired properties and application of the gel. For example, if it is an alcoholic solution, the drying process is done by burning alcohol. After the drying stage, the produced gels are powdered and then calcined. The sol-gel method is a cost-effective method and due to the low reaction temperature there is good control over the chemical composition of the products. The sol-gel method can be used in the process of making ceramics as a molding material and can be used as an intermediate between thin films of metal oxides in various applications. The materials obtained from the sol-gel method are used in various optical, electronic, energy, surface engineering, biosensors, and pharmaceutical and separation technologies (such as chromatography). The sol-gel method is a conventional and industrial method for the synthesis of nanoparticles with different chemical composition. The basis of the sol-gel method is the production of a homogeneous sol from the precursors and its conversion into a gel. The solvent in the gel is then removed from the gel structure and the remaining gel is dried. The properties of the dried gel depend significantly on the drying method. In other words, the “removing solvent method” is selected according to the application in which the gel will be used. Dried gels in various ways are used in industries such as surface coating, building insulation, and the production of special clothing. It is worth mentioning that, by grinding the gel by special mills, it is possible to achieve nanoparticles.

1. Introduction

Today, various methods such as sol-gel method (solution method), vapor phase compression method, mechanical alloying method or collision with high-energy pellets, plasma method, and electrochemical methods are used for the production and synthesis of nanoparticles. Although all the mentioned methods have the ability of producing large volumes of nanomaterial, the sol-gel method has a higher popularity and industrial application than other existing methods [1–5]. Due to its unique properties and characteristics, this method is capable of producing high quality
nanoparticles of the same size on an industrial scale [6–8]. This method is capable of producing two or more types of nanoparticles simultaneously, meaning that alloy products are synthesized in one step by mixing two or more metal (or metal oxide) precursors in certain proportions [9–11]. Of course, there are other methods such as plasma method and electrochemical methods that have the ability of synthesizing alloy products in one step, but their main difference with sol-gel method is the industrial scale of sol-gel method [12–15]. In addition, the sol-gel method makes it possible to make highly homogeneous composites with very high purity (99.99% purity) [16–20]. Another advantage of this method compared to conventional methods is the lower temperature of the process in it, so that the production of metal and ceramic nanomaterial with this method is possible in the temperature range between 70 and 320°C [21–24]. The other methods mentioned produce nanomaterial in the temperature range of 1400–3600°C [25–28]. The sol-gel process is a bottom-up synthesis method. In this process, the final products are formed by performing a number of irreversible chemical reactions [29–31]. During these reactions, the primary homogeneous molecules (sol) become an infinite, heavy, three-dimensional molecule called a gel [32–35]. The conversion of tuberculosis to gel is done through a process called “compaction process” and leads to the production of wet gel [36–38]. Figure 1 shows an overview of the different stages of sol-gel process from precursor to aerogel. For the synthesis of binary or tertiary hybrid systems, a mixture of salts with different chemical compositions is used [39–41]. Each of the primary salts has a unique reaction rate [42–45]. The rate of reaction of salts depends on various factors such as pH, concentration, type of solvent, and temperature [46, 47]. The polymer gel formed from the density of the cell is a three-dimensional structure and is formed by joining the cavities [48–50]. After drying the gel, due to volumetric shrinkage, a solid and rigid structure is obtained [51]. It should be noted that, by controlling the drying conditions of the gel, it is possible to achieve nanosized porosity [52, 53]. The advantage of nanoporosity is the very large specific surface area compared to normal porosity [54–56]. For example, nanoporous carbon materials are used to store hydrogen, so that hydrogen is trapped and stored in the porosity of the carbon material [57–61]. Figure 2 shows the SEM image of these nanoporous materials. In the sol-gel process, the conversion of sol to gel is usually done by changing the pH or changing the concentration of the solution [62–67]. The main advantages of the sol-gel process are the high purity of the product, the narrow particle size distribution, and the achievement of uniform nanostructure at low temperatures. This method is commonly used to synthesize metal nanoxides [68–70]. As mentioned, the sol-gel process involves changing the state from sol to gel using a variety of techniques; most of which use gentle drying to remove the solvent [71–74]. Since the drying of the gel is accompanied by its shrinkage, providing suitable conditions to prevent the formation of cracks is one of the most important issues in this process [75–78]. The produced gel has the ability of casting and by molding and drying it is possible to produce integrated pieces [79–81]. Products from casting or molding are used as filters or membranes. It is possible to create thin films with a thickness of 50–500 nm by sol-gel method [79, 82–84]. For this purpose, different coating methods can be used: (a) dipping coating method and spin coating, (b) spray coating process, (c) flow coating process, (d) capillary coating process, and (e) the climbing cover process [85–88]. Sol-gel thin films have a wide range of applications in the electronics and chemical industries [89]. In addition, coatings obtained from the sol-gel process also affect the optical properties of the material. Figure 3 shows an overview of the various sol-gel coating processes [90–94]. The sol-gel method can be used in the production of composite or nanocomposite materials [95–97]. For this purpose, continuous porosity at the nanoscale is used as a place for loading secondary materials [98–100]. Loading means adding a substance into the cavities. This is done using methods such as purification in the molten phase or chemical reactions. Nanoporous materials are classified into nanocomposites [101–104]. One of the applications of nanoporous materials is their use in catalytic industries [105, 106]. The pores of nanoporous materials are filled with a variety of industrial catalysts, and due to the very large active surface area of these materials the catalytic efficiency increases and the cost of the product decreases [107–109]. Figure 4 shows a diagram of the loading of secondary particles into the nanoporous material [110–114]. In order to produce denser parts, the synthesized or produced parts are subjected to sintering operations [115]. The high specific surface area increases the rate of compaction or compaction of the structure, so the nanoporous gels compact better and faster during the sintering process [116–119]. On the other hand, it should be noted that increasing the temperature during this process causes the growth of grains and creates a microstructure consisting of coarse grains [120–123]. Figure 5 shows an overview of the types of processes that can be performed by the sol-gel method and the products derived from them.

2. Aerogels

Aerogels are gels with nanometer pores, low density, porosity, and high internal area. Due to these properties, aerogels are widely used in applications that require sound insulation as well as high light transmittance. The aerogel synthesis process consists of two stages; in the first stage or the gel-making stage, the solvent penetrates the gel and in the next stage the solvent is removed or dried. In general, the term aerogel refers more to the internal structure of a substance than to its constituent materials; therefore, it is possible to synthesize aerogels by a wide range of raw materials with different chemical compositions [124–127]. The raw materials used to synthesize aerogels include minerals, organic, and composite. Aerogels were first introduced in 1931 by Cuce et al. [76–78]. The chemical composition of the first aerogel introduced was silica. The word “aerogel” is made up of the words air and gel. In general, aerogel is a very light, porous substance derived from a gel. In aerogel synthesis, the liquid phase of the gel is replaced by gas (air), which results in a material with low
density and thermal conductivity [79, 80]. About 99.8% of this nanomaterial is composed of air, so their structure is solidly porous with a network of porous materials in the form of gas packets, which makes the aerogels almost weightless. Aerogels are known to be the lightest and least dense solids, with about 50–99.5% of their volume being air [77, 78, 128–130]. These gels have a surface area of 250–3000 square meters per gram so that the surface of an aerogel with a volume of one cubic inch is about a football field [81]. Aerogels usually have amorphous structures and lack crystalline order in their microstructure. For this reason, these nanomaterials are very brittle and have very high transparency [82].

2.1. Properties of Aerogels

2.1.1. Thermal Conductivity Aerogels. Aerogels are known as the best thermal insulation material. The main reason for the low thermal conductivity of these nanomaterials is their porous structure, in such a way that the porosity in their structure prevents continuous and proper flow of heat. Another reason for the insulation of aerogels is the limited heat transfer mechanisms in them. In general, there are three mechanisms for heat transfer, radiation, convection, and conduction. Aerogels neutralize the two mechanisms of convection and conduction in heat transfer [82]. The main reason for the elimination of the conduction mechanism is the presence of excessive gas porosity in the structure of aerogels and the reason for the elimination of the convection mechanism is the inability of air of circulating throughout their crystal lattice [131–134]. However, heat transfer by radiation is possible due to the possibility of infrared waves (these waves have the ability of transferring heat) through the aerogels. Silica aerogels are used in the manufacture of insulated windows due to their low heat transfer. The thermal conductivity of aerogels is even lower than that of their constituent gases. This effect occurs when the size of the gas cavities is approximately equal to their mean free path [83].

Yang et al. [134] concluded, in an experimental study on the thermal conductivity of aerogel-enhanced insulating materials under various hygrothermal environments, as
nanostructured material, that aerogel insulation materials are widely used due to their outstanding thermal conductivity. To improve the hygrothermal performance of aerogel-based insulation materials, various aerogel-enhanced insulating materials were exploited, and their pore distributions and hygrothermal performance under different moisture

Figure 3: Scheme of synthesis of samples by sol-gel method: (a) films synthesized from colloidal sol and (b) powder synthesized from gel [1–5].

Figure 4: Image of porous nanomaterial that after modification of the surface, the desired catalyst is loaded into its cavities [1].
contents, environmental temperatures, and moisture levels were analyzed and evaluated. The results show that (1) the higher the content of aerogel, the lower the density and the thermal conductivity of an aerogel-enhanced HGB, and a 64% aerogel-enhanced HGB had the optimal thermal performance and density \( (\lambda \sim 0.0465 \text{ W/m}\cdot\text{K}, \rho \sim 233.35 \text{ kg/m}^3 \text{ at } 20^\circ\text{C}) \) in the dry state; (2) a power function relation was adopted to fit the thermal conductivity and mass MC of the specimens, and with increasing aerogel content the greater the MC was, the slower the specimens' thermal conductivity increased; (3) the humidity-driven changes in the thermal conductivity of aerogel-enhanced materials are significantly greater than temperature-driven changes. For example, as the temperature increased from 20 to 70°C, the thermal conductivity increased linearly (high correlation) in the range of 9.28–13.97%; however, when the RH increased from 0% to 98% at 35°C, the increase in the coefficient of thermal conductivity of the 64% aerogel-enhanced HGB was the highest (61.54%); (4) the higher the content of aerogels was, the more sensitive the humidity was to the thermal conductivity of the materials.

2.1.2. Hydrophobicity of Aerogels. In general, aerogels are composed of particles with a diameter of 2–5 nm. After the aerogel is synthesized, large amounts of hydroxyl groups are formed on its surface. These groups cause the aerogel to react with water and cause it to dissolve severely in it. One way to make hydrophilic aerogels hydrophobic is to replace their hydroxyl (-OH) group with nonpolar (-OR) groups \([76, 80, 135, 136]\). Therefore, it can be concluded that aerogels are inherently hydrophilic, but they can be dehydrated by chemical operations. In these groups, \( R \) indicates aliphatic compounds. To hydrophobicize silica aerogels, these structures are synthesized from methyltrimethoxysilane and tetraethoxysilane in play conditions and dried in supercritical methanol. In this method, the drying process is performed at high temperatures and leads to hydrophobicity of the surface of the aerogels. Figure 6 shows an overview of the synthesis steps of hydrophobic silica aerogels. Aerogels with at least 20% methyl methotrexate remain floating on the water, so they are considered hydrophobic. Figure 7 shows the structural differences between hydrophobic and hydrophilic silica aerogels \([76–83]\).

Li et al. \([135]\) concluded that hydrophobic silica aerogels were produced by the \(-\text{Si}(\text{Me})_3\) (trimethylsilyl substituent: TMS) modification of alcogels followed by \(\text{CO}_2\) supercritical drying. The structure of trimethylsilyl modified silica aerogel (TMSA) was the silica matrix produced by hydrolysis and condensation of only tetramethoxysilane (TMOS). TMS was modified on the surface of the silica matrix. TMSA was extremely moisture-resistant. The density, size, and transparency of TMSA samples were maintained after the moisture-resistance test. The moisture resistance of trimethylsilyl modified silica aerogels (TMSAs) was superior to that of conventional aerogels supercritically dried by the ethanol method. Another feature of TMSA was the small shrinkage during supercritical drying. The shrinkage ratio of TMSA was less than 3% by comparison with 5% for conventional aerogels. TMSA had the same transparency as conventional aerogels, with a transmittance of 90% at a thickness of 1 cm. The existence of TMS was observed using infrared, \(^{13}\text{C}\) nuclear magnetic resonance (NMR) and \(^{29}\text{Si}\) NMR.

2.1.3. Mechanical Properties of Aerogels. One of the main features of aerogels is their lightness and brittleness. However, they are strong enough to be carried by hand. In general, the compressive strength, tensile strength, and elasticity of these materials are very low. The mechanical properties of aerogels are highly dependent on their
structural conditions such as the quality of cavity cohesion and density. One way to increase the structural integrity of aerogels and improve their mechanical properties is to place the gels in alkoxide solutions during the aging process. In this way, the gels become more resistant to capillary pressure [76–83]. Figure 8 shows the techniques for characterizing the mechanical properties of aerogels.

Feng et al. [136] in a study of mechanical properties and thermal conductivity of aerogel-incorporated alkali-activated slag mortars concluded that compressive strength, thermal conductivity coefficient, and porosimetric properties of alkali-activated slag (AAS) mortars containing silica aerogel were investigated experimentally in this study. For this purpose, slag mortar mixtures at 0.75% and 1.0% aerogel content ratios were prepared, and these mortar mixtures were activated with lithium carbonate (Li₂CO₃) at 0.03% and 1.50% dosage rates. Mortar samples were exposed to curing process in water for 2, 7, and 28 days, and the samples, which completed the curing stage, were subjected to the compressive strength test. The porosimetry test and the thermal conductivity coefficient measurement were carried out following the compressive strength test on 28-day samples. The varying aerogel content rate in the mixtures and the effects of the dosage of Li₂CO₃ on the gel, capillary, and macropore distributions and the effect of changing porosimetric properties on compressive strength and thermal conductivity coefficient were analyzed in detail. Experimental studies have shown that AAS mortars including an optimum 0.75% aerogel content rate and 0.03% Li₂CO₃ activation provided a compressive strength of 34.1 MPa and a thermal conductivity coefficient of 1.32 W/m·K. Aerogel addition provides a partial compressive strength increase at 7- and 28-day samples while it also causes maximum strength loss of 5.0% at 2-day samples.
3. Methods of Synthesis of Aerogels

In general, the methods of synthesis of aerogels are based on their production by polymerization reactions of various compounds in the form of gels. The wet gels produced are then dried by various methods. The final product is a dry material with a very porous and light texture [137–139].

3.1. Sol-Gel Process. The sol-gel process is performed at low temperatures (usually less than 100°C) and in the liquid state. Of course, the final product is solid, and these solids are formed as a result of the polymerization process, which involves the establishment of M-OH-M or M-O-M (where M represents the metal atom) between the metal atoms in the raw materials. The synthesis of aerogels using the sol-gel process consists of two steps, which are as follows [140–143]:

(i) The first stage involves the formation of separate colloidal solid particles with nanometer dimensions.
(ii) The second stage involves colloidal particles in the solvent joining together to form a gel.

Figure 9 shows an overview of the process of synthesizing silica aerogels using the sol-gel process [76–83].

3.2. Chemistry of Sol-Gel Process. There are several parameters which influence the hydrolysis and condensation reactions (sol-gel process), including the activity of the metal alkoxide, the water/alkoxide ratio, solution pH, temperature, nature of the solvent, and additive used. Another consideration is that catalysts are frequently added to control the rate and the extent of hydrolysis and condensation reactions. By varying these processing parameters, materials with different microstructures and surface chemistry can be obtained. Further processing of the “sol” enables the fabrication of ceramic materials in different forms. Thin films can be produced on a piece of substrate by spin coating or dipping. When the “sol” is cast into a mold, a wet “gel” will form. With further drying and heat treatment, the “gel” is converted into dense ceramic or glass particles. If the liquid in a wet “gel” is removed under supercritical conditions, a very porous and light texture aerogels is obtained.

3.3. Precursors for Sol-Gel Processing. The precursor is nothing but the starting material for the sol-gel process.

1. Precursors should be soluble in the reaction media
2. They should be reactive enough to participate in the gel forming process [20]

Some salts, oxides, hydroxides, complexes, alkoxides, acrylates, and amines are used as precursors if soluble in proper solvents [21, 22]. Alkoxydes are the most common sol-gel precursor, since they are commonly available. Bradley et al. have well explained the basic chemistry of the precursor [23]. It is very difficult to predict the type of precursor to be used for a given purpose. The reactivity of precursor depends not only on its chemical nature but also on the applied reaction condition [24]. Compared to the precursors of other elements, the network forming power of Si is more to build up a gel [18]. That is why other expensive alkoxide precursors can be substituted by cheaper ones like silicon alkoxide such as TEOS and TMOS and water-soluble precursor such as Na₂SiO₃ for sol-gel processing.

3.4. Methods of Converting Wet Gel to Aerogel

3.4.1. Drying by Supercritical Method. This method involves heating wet gels in a closed space with controlled pressure. Of course, the temperature and pressure of this medium must be adjusted so that these values are in the critical area of the liquid trapped in the pores of the gel [80, 82, 144–147]. As a result, the solvent can be removed from the system as gas. Figure 10 shows how to find the critical point using pressure-temperature diagrams, and Figure 11 shows a diagram of an autoclave used for supercritical drying.

Supercritical solvents are similar to both liquids and gases in that they are dispersed like gases and have the same density and thermal conductivity as liquids. These solvents usually have high pressure and heat. Also, supercritical solvents have less surface tension than liquids, which preserves the porous structure of aerogels and prevents their structure from collapsing during the drying process [76, 83, 148–150]. Figure 12 shows the names and properties of some common supercritical fluids.

3.4.2. Drying Methods. It is possible to dry organic gels produced in organic solvents by using solvent evaporation at ambient pressure (without severe contraction). Also, in these conditions, surfactants can be used to reduce capillary pressure. Other common drying methods include the following [76–83]:

(i) Use of chemical additives that control the drying process such as glycerol, formaldehyde, oxalic acid, and tetramethylammonium hydroxide;
(ii) Drying processes at ambient pressure: these methods can be used on an industrial scale;
(iii) Freeze drying method: the liquid inside the gel is first frozen and then dried by sublimation. The material obtained from this method is called Cryogel.

Xylogels are substances that have dried under normal conditions. These materials are not much different from aerogels and the main difference between them is the number and size of cavities, as the aerogels have more and larger cavities due to drying by supercritical method [151–153]. Figure 13 shows a flowchart of the synthesis steps of aerogels, carbogels, and subgels.
3.4.3. Types of Aerogels.

Aerogels are classified into four groups according to the chemical composition of their gels: inorganic aerogels including silica aerogels and alumina aerogels, organic aerogels, carbon aerogels, and hybrid aerogels [76, 77].

(1) Inorganic Aerogels. Silica gels were the first mineral gels synthesized under acidic conditions. Of course, natural organic gels are more common today. It is possible to convert all metals or semiconductor oxides to gels. The most common mineral aerogels are silica aerogels, titanium, zirconium, tin, aluminum, vanadium, chromium, iron, tantalum, molybdenum, and niobium-based aerogels. Binary or ternary oxide aerogels can also be synthesized. Of course, these aerogels are very fragile due to their ceramic nature [78–81].

(2) Organic Aerogels. Organic aerogels are synthesized using organic precursors, which produce strong polymer covalently bonded (C-C) organic polymers. The most common organic compounds used to synthesize these aerogels are resorcinol-formaldehyde and melamine-formaldehyde (resin-formaldehyde). Condensation of the mentioned organic compounds in alkaline aqueous solution is used for the synthesis of organic aerogels. The alkaline aqueous solution usually used is sodium hydroxide, in which sodium hydrogen carbonate is used as a catalyst [82, 154–159]. Figure 14 shows the melamine-formaldehyde density reaction for the synthesis of organic aerogels.

(3) Carbon Aerogels. Carbon aerogels were first synthesized in 1990. A common method for synthesizing these aerogels is the pyrolysis of organic aerogels at temperatures above 500°C, as a result of which the organic aerogels become electrically conductive carbon air gel. During the thermocouple process, the area and amount of pores and pores of the carbon air gel are preserved. Studies show that the pyrolysis process at temperatures below 1000°C increases the number of microspores, so that the largest surface area...
| S.No | Compound          | Critical temperature (°C) | Critical pressure (bar) |
|------|-------------------|---------------------------|-------------------------|
| 1    | Carbon dioxide    | 31.3                      | 72.9                    |
| 2    | Ammonia           | 132.4                     | 112.5                   |
| 3    | Water             | 374.15                    | 218.3                   |
| 4    | Nitrous oxide     | 36.5                      | 71.7                    |
| 5    | Xenon             | 16.6                      | 57.6                    |
| 6    | Krypton           | -63.8                     | 54.3                    |
| 7    | Methane           | -82.1                     | 45.8                    |
| 8    | Ethane            | 32.28                     | 48.1                    |
| 9    | Ethylene          | 9.21                      | 49.7                    |
| 10   | Propane           | 96.67                     | 41.9                    |
| 11   | Pentane           | 196.6                     | 33.3                    |
| 12   | Methanol          | 240.5                     | 78.9                    |

**Figure 12:** Names and properties of some common supercritical fluids [79].

![Flow diagram](image1)

**Figure 13:** (a) A flow diagram depicting the aerogel preparation process; (b) a schematic depicting the in situ synthesis of Ni within the aerogel, which corresponds to the final two stages in the process (a) [79].
of these aerogels is obtained at a temperature of 600°C. As the temperature gradually increases, the amount of cavities decreases and at temperatures above 2100°C the volume of cavities reaches zero, which indicates the formation of closed cavities. Carbon aerogels are synthesized in various forms such as monoliths, powders, films, and granules, the properties of which are shown in Figure 15 [76, 83, 160, 161].

(4) Hybrid Aerogels. The synthesis of aerogels using organic-mineral compounds, also known as hybrid aerogels, significantly increases the applications of aerogels. For example, with the introduction of organic structures into the silica surface, their hydrophobic and elastic properties are improved. Hybrid aerogels are usually synthesized by the sol-gel method. One of the main advantages of hybrid aerogels over other aerogels is their lower fragility [149].

4. Advantages of Sol-Gel Method

(i) Simplicity of the process
(ii) Preparation of high purity products;
(iii) Very high production efficiency
(iv) Production of optical components with complex shapes
(v) Synthesis of uniform compounds in the form of composite oxides
(vi) Ability of designing and controlling chemical composition and obtaining a homogeneous composition
(vii) Ability of using the product with special shapes such as fibers and aerogels
(viii) Surface coverage
(ix) Ability of using this process to synthesize amorphous materials in thin layers
(x) Production of materials with modified physical properties, such as low thermal expansion coefficient, low UV absorption, and high optical transparency
(xi) Production of porous and rich materials with organic and polymeric compounds
(xii) High chemical reactivity of precursors due to process in solution phase
(xiii) Precise control of material structure with the possibility of adjusting the variables of the early stages of tuberculosis and network formation
(xiv) Low initial investment while having high quality products

The sol-gel process is a common and almost old method [161]. In the mid-nineteenth century, the tendency to use the sol-gel process to synthesize mineral ceramics and glass materials began with the efforts of the likes of Ebelman and Graham and the study of silica gels. In the 1950s, extensive studies began on the synthesis of ceramics and glass structures using the sol-gel method. This method has the ability of synthesizing many mineral oxides such as TiO₂, SiO₂, and ZrO₂. Depending on the drying conditions of the wet gel, two types of dry gels are obtained, aerogels and xerogels. By definition, an aerogel is a dry gel obtained by removing moisture from a wet gel. Depending on the moisture removal method, the structure of this gel largely preserves the structure of the wet gel (primary gel). Wet gel is the gel that results from the conversion of tuberculosis to gel during the compaction process. The sol-gel process involves the transfer from the liquid phase of the “cell” (colloidal solution) to the “gel” phase. Figure 16 shows the types of gel processing methods and techniques presented to turn it into products. As can be seen, the products can be produced both in powder and thin layers and in porous or dense materials [76–83].

Inorganic metal salts or organic metal compounds such as metal alkoxides are commonly used as precursors. Colloidal suspension or “cell” is formed after a series of hydrolysis reactions and condensation of precursors. The TB particles then condense into a continuous liquid (gel) phase. With additional drying and heat treatment, the gel becomes a dense glass or ceramic material. In general, three reactions are used to describe the sludge process: (1) hydrolysis, (2) alcohol condensation, and (3) water condensation. Figure 17 shows the general steps of the sol-gel process and the types of synthesized materials. Because water and alkoxides are incompatible, alcohols are commonly used as a solvent. Due to the presence of a solvent, the cellulose and alkoxide precursors mix well with the water and facilitate the hydrolysis process. Figure 18 shows the hydrolysis and compaction reactions (in both water and alcohol) in the sol-gel process to produce silica gel.

During the hydrolysis reaction, by adding water to the alkoxide groups (OR), these groups replace the hydroxyl
The next condensation reaction involves the silanol (Si-OH) group, which produces siloxane (Si-O-Si) bonds with by-products including water (condensed water) or alcohol (condensed alcohol). As the number of siloxane groups increases, the nanoparticles begin to bridge with each other to form a silica network. As soon as the wet gel dries, the solutes trapped in the network are released. With additional heat treatment at high temperatures, the organic waste in the structure is removed, destroying the interconnected cavities and eventually forming compressed glass or ceramics. Figure 19 shows the different gel states including wet gel, dry gel, and aerogel [76–83].
**Precursor**
(usually mixture of metal, bimetallic or \(\mu\)-oxobimetallic alkoxide)

- **Hydrolysis**
- **Sol**
- **Condensation**
- **Gel**
- **Critical Point Drying**
- **Melt at lower temperature**
- **Speciality Phase**
- **Noncrystalline Ceramics**
  (Xerogel, Aerogels)

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**Figure 17**: An overview of the various stages of the sol-gel process and the types of synthesized products [76].

**Step 1: hydrolysis**

\[
\text{OR} \quad \text{OR} + \text{H}_2\text{O} \leftrightarrow \text{OR} \quad \text{OH} + \text{R-OH}
\]

**Step 2: condensation**

- **(a) Water condensation**

\[
\text{OR} \quad \text{OH} + \text{OH} \quad \text{OR} \leftrightarrow \text{OR} \quad \text{O-} \quad \text{OR} + \text{H}_2\text{O}
\]

- **(b) Alcohol condensation**

\[
\text{OR} \quad \text{OH} + \text{OR} \quad \text{OR} \leftrightarrow \text{OR} \quad \text{O-} \quad \text{OR} + \text{R-OH}
\]

Where R is an alkyl chain. For TEOS, R = \(-\text{CH}_2\text{CH}_3\)

**Figure 18**: Hydrolysis reactions, condensation with water and alcohol to produce TB, and finally synthesis of silica gel [76].

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**Springback phenomena**

**Figure 19**: Different states of the gel after the sol-gel process: wet gel, dry gel, and aerogel, respectively.
5. Sol-Gel Process Steps

5.1. Homogeneous Solution. In the sol-gel process, we seek to produce a homogeneous solution of precursors and alcohols. For this purpose, first the solvent (usually containing water, alcohol, organic solvents, or a proportion of them) and the precursor are mixed in a container to obtain a homogeneous solution. To achieve a homogeneous solution, a mixture of two solvents in specific concentrations is often used to fully dissolve the precursors. For example, some metallic organic precursors must first be dissolved in a water-soluble organic solvent and then the resulting solution is dissolved in water. However, in cases where the precursor is metal salt, it is soluble directly in water and does not require an organic solvent. Although alkoxide precursors are more widely used, the sol-gel process can be performed in other ways, in which, instead of homogeneous tuberculosis, a "relatively stable colloid" is used for gel formation. Metal alkoxides, as precursors used in the sol-gel method, are a class of organic metal compounds consisting of an organic base attached to a metallic or quasimetallic element. As an interesting example, consider silicon tetroxide (\(\text{Si} \left(\text{OC}_2\text{H}_5\right)_4\)). This precursor is also known as tetrathoxysilane (TEOS). Table 1 lists some common precursors for the synthesis of metal oxides using the sol-gel method and their functional groups [76–83].

5.2. Sol Formation. After forming a homogeneous solution, it should be converted to tuberculosis. This phase is built around the hydrolysis reaction. The word “hydrolysis” is a combination of the words “hydro” and “lysis” and means water decomposition. The term decomposition means that a complex compound becomes a simpler compound [37–39]. In chemistry, water can sometimes break down a molecule into simpler molecules. In general, reactions in which the molecules are broken down by chemical reactions with water are called hydrolysis. Figure 20 shows an overview of the steps of the sol-gel method, including the formation of a homogeneous solution, the formation of the sol, the conversion of the sol-gel, and the removal of the solvent from the gel (drying). In the hydrolysis reaction, large molecules are broken down into simpler molecules with water. The presence of water causes a hydrolysis reaction on the precursor and somehow activates it so that the metal oxide particles combine to form fine and solid particles dispersed in the solvent [40–45]. In a real solution, the solute is dispersed uniformly in the solvent as an atom, molecule, or ion, and the particle size does not exceed 1 nm. If the particle size of the solvent is greater than 100 nm, the particles gradually settle to form a suspension mixture [46–50]. If the particle size varies between 1 and 100 nanometers, they usually remain scattered throughout the mixture, which is called colloid. The sol consists of very fine particles (less than 100 nm) dispersed in the solvent phase and are, in fact, a solution or more precisely a colloidal mixture [51–54]. Thus, according to the above explanations, a comprehensive definition of tuberculosis can be given; the most important thing to say about a good quality TB is that the resulting TB must be prepared in such a way that it can be stable for months and not settled. In other words, the particle size must be small enough that the Brownian motion of the particles overcomes the force of gravity and prevents them from settling. In this way, the resulting mixture remains homogeneous for a long time. If TB has such characteristics, it can be hoped that a homogeneous, pure, and high-yield product will be produced [55–60].

5.3. Gel Formation. In order to form a gel, it is sufficient to stimulate the solution in some way, so that the dispersed fine particles (each containing several molecular or atomic units of the corresponding precursors) begin to collect [84–87]. By creating physical and chemical interactions between suspended particles and those dispersed in the tuberculosis solution, units of tens of thousands of molecules line up together to form an infinitely large three-dimensional molecule that occupies the entire volume of the reaction vessel [61, 65, 88, 89]. Figure 21 shows the compaction reaction for the synthesis of silica gel from a cell. This giant molecule, which has many pores and traps all the solvent inside it, is called wet gel. Figure 22 shows an overview of the sol-gel process to produce a uniform, dry gel.

As mentioned, wet gel production requires stimulation of the tuberculosis solution. This stimulation can be done using a suitable reagent (pure water or water with NaCl and NaOH). In fact, the cell-to-gel conversion phase, controlled by reactions called condensation, is an inorganic polymerization reaction whose end product is an oxide network containing MOM metal oxide clusters [66–70]. The compaction reaction is exactly the opposite of the hydrolysis reaction. In the hydrolysis reaction, large molecules are converted into simpler components by consuming water, but in the compaction reaction two simple molecules combine to form a more complex molecule. As the molecules combine in the compaction reaction, a small molecule such as water is released. A compaction reaction is possible when two hydroxides (or one hydroxide + one metal alkoxide) (M-OR + HO-M) combine to form a metal oxide (M-O-M) [71–73]. Therefore, the gel produced will be soluble and in order to complete the sol-gel process and achieve a dry gel, the solvent must be separated using some methods. The produced gels have different types depending on the solvent used or the drying method and show various properties and applications [74, 75].

6. Application of the Nanomaterials Prepared by Sol-Gel Method

(i) Synthesis at low temperature
(ii) Preparation of high purity products
(iii) Very high production efficiency
(iv) Production of optical components with complex shapes
(v) Synthesis of uniform compounds in the form of composite oxides
(vi) Possibility of designing chemical composition and obtaining homogeneous composition

(vii) Ability of using the product in special forms such as fibers, aerogels, and surface coatings

(viii) Ability of using this process to synthesize materials in an amorphous state and applying them to thin layers

(ix) Production of materials with modified physical properties such as low thermal expansion coefficient and low UV absorption and high optical transparency

(x) Production of porous materials that allow enrichment with organic and polymeric compounds

(xi) High chemical reactivity of precursors due to process in solution phase

(xii) Precise control of material structure with the possibility of adjusting the variables of the initial stage of tuberculosis and network formation low
initial investment and high quality products
[134, 135]

7. Prospective for the Nanomaterials Prepared by Sol-Gel Method

Sol-gel chemistry offers a flexible approach for obtaining a diverse range of materials. It allows differing chemistries to be achieved as well as offering the ability of producing a wide range of nano-/microstructures. The paper commences with a generalized description of the various sol-gel methods available and how these chemistries control the bulk properties of the end products. Following this comes a more detailed description of the biomedical areas where sol-gel materials have been explored and found to hold significant potential. One of the interesting fields that has been developed recently relates to hybrid materials that utilize sol-gel chemistry to achieve unusual composite properties. Another intriguing feature of sol-gels is the unusual morphologies that are achievable at the micro- and nanoscale. Subsequently, the ability of controlling pore chemistry at a number of different length scales and geometries has proven to be a fruitful area of exploitation that provides excellent bioactivity and attracts cellular responses and enables the entrapment of biologically active molecules and their controllable release for therapeutic action. The approaches of fine-tuning surface chemistry and the combination with other nanomaterials have also enabled targeting of specific cell and tissue types for drug delivery with imaging capacity.

8. Conclusion

(1) The sol-gel process is a bottom-up synthesis method. In this process, the final products are formed by performing a number of irreversible chemical reactions. In this paper, the selection of suitable catalysts, the operating mechanism of the catalysts, and the drying steps of the wet gel were investigated. It was said that the reaction rate between water and alkoxides is very low. With the addition of alcohol, the solution becomes more dilute and the reaction rate decreases sharply. Therefore, in order to increase the reaction rate in the sol-gel process, a catalyst is used. It was emphasized that the catalysts used in the sol-gel process should be selected in such a way that it can be easily removed from the environment after the reaction. As a comprehensive example, the mechanism of action of acidic and alkaline catalysts in the silica gel sol-gel process was investigated. It has been said that the use of acidic or alkaline catalysts increases the rate of the hydrolysis reaction and the amount of sol produced. It was emphasized that since the solvent is one of the internal components of the gel and is responsible for forming its network, it is very difficult to maintain the structure of the cavities by removing the solvent. In order to maintain the integrity of the gel structure, it is necessary to allow some time before drying for the bonds between the particles to become stronger and stronger. Two methods for solvent separation from gels were introduced. In the first method, the gel is placed in the atmosphere after production to dry spontaneously. The gel obtained from this method is called aerogel. In the second method, called the supercritical method, the changes in the solid network are minimized. The material obtained from this method has porous networks and low strength and is “hollow” in correction. This product is called aerogel.
Finally, based on what has been said, it is possible to control the final structure and properties of the products obtained from the sol-gel process by precisely controlling the variables related to wet gel drying, sol, and gel synthesis.

(2) Aerogels are gels with nanometer pores, low density, porosity, and high internal area. In this article, different properties, synthesis methods, and types of aerogels and their applications were studied. Aerogels are said to be the lightest and least dense solids, so that about 50–95.5% of its volume is air. It was emphasized that aerogels are known as the best thermal insulation material. The main reason for the low thermal conductivity of these nanomaterials is their porous structure, so that the porosity in their structure prevents continuous and proper flow of heat. Aerogels neutralize the two mechanisms of convection and conduction in heat transfer. It was pointed out that the transparency of aerogels is one of their important physical properties and plays an important role in the application of these nanomaterials. Solutions were also proposed to improve the mechanical properties of these materials. Aerogels have been said to be inherently hydrophilic but can be dehydrated by chemical treatment. It was pointed out that aerogels are light and brittle and their mechanical properties are strongly dependent on their structural conditions such as the quality of the cavities and their density. It was emphasized that the methods of synthesis of aerogels are based on their production by polymerization reactions of different compounds in the form of gels. The wet gels produced are then dried by various methods. The sol-gel method was introduced as the most common method of synthesizing aerogels. Then, wet gel drying methods such as supercritical drying method were introduced. Aerogels are said to be divided into four groups: mineral, organic, composite, and hybrid, according to the chemical composition of their gels. Then, important applications of aerogels such as those in electronics and energy equipment, those as catalysts, building materials, shock absorbers, and sensors were introduced.

Conflicts of Interest

The authors declare that they have no conflicts of interest.

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