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Introductory Chapter: A Brief Semblance of the Sol-Gel Method in Research

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Additional information is available at the end of the chapter

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1. Introduction

The emergence of the sol-gel process occurred in the year 1921. In the 1960s, its development was given due to the need of new synthesis methods in the nuclear industry. This development began to become popular around 1984 and reached its splendor in 2011 as shown in **Figure 1** [1]. Dr. Jeffrey Brinker is a pioneer in the synthesis of materials and sol-gel-science and sets an

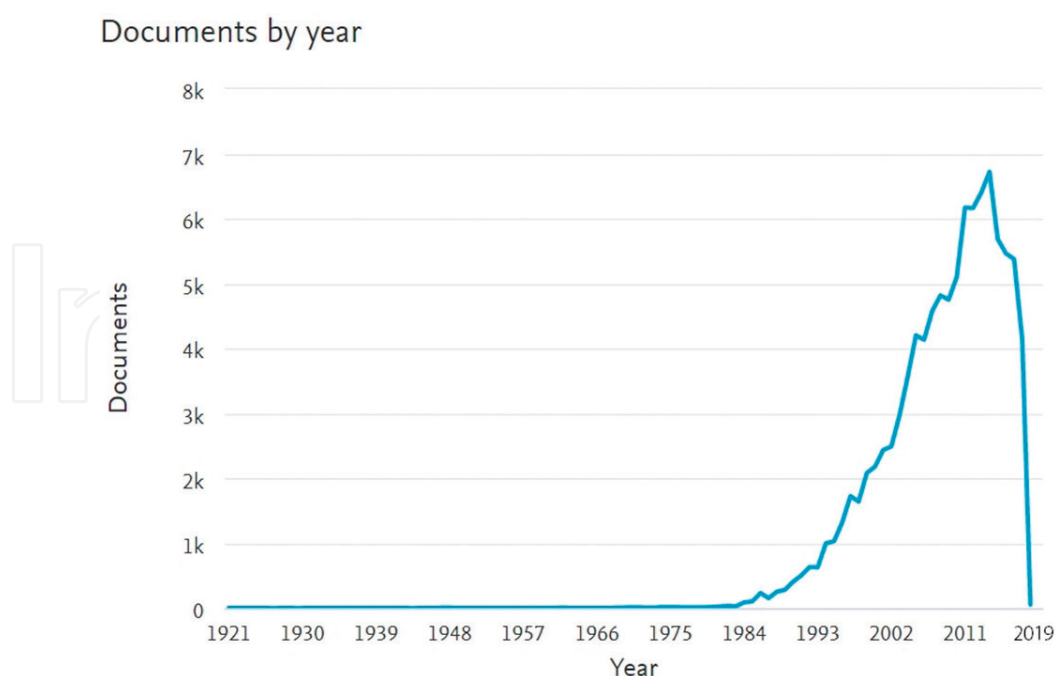


Figure 1. Temporal evolution of sol-gel publications.

important guideline for the flowering of the sol-gel method [2]. Other researchers who have developed their research in the sol-gel field making substantial and important contributions to this field are Dongyuan Zhao and David Avnir. The method of sol-gel is a route of synthesis more used worldwide that there is a meeting named International Sol-Gel Conference [3], which is held every 2 years, bringing together renowned scientists with the new generations, who give contributions of their work in different areas, all linked to the sol-gel method.

2. General mechanism

A description of the sol-gel process can be formation of an oxide network through polycondensation reactions of a molecular precursor in a liquid.

In general, in this process, several stages are identified, starting with a silicate solution and then forming a sol, which will then be transformed into a gel, and finally, a dry gel is obtained which is generally formed by a three-dimensional network of silica, with numerous pores of various sizes interconnected. **Figure 2** presents an outline of the routes of this mechanism.

Among the advantages of using the sol-gel process in the synthesis is because it can be carried out at room temperature, it allows us to produce a wide range of novel and functional materials, with potential applications in different areas; and finally, it is really attractive compared to other methods, due to its low production costs.

Sol-gel samples can be designed with a wide variety of morphologies, such as monoliths, films, fibers, and powders. In particular, films are the most important from the technological point of view.

The process begins with the formation of a “sol,” which is a stable dispersion of colloidal particles (amorphous or crystalline) or polymers in a solvent. A “gel” is formed by a three-dimensional continuous network, which contains a liquid phase, or by the joining of polymer chains. In a colloidal gel, the network is built from agglomerates of colloidal particles. While in a polymer gel, the particles have a polymeric substructure composed of aggregates of sub-colloidal particles. Generally, van der Waals forces or hydrogen bonds dominate the interactions between the sol's particles. During synthesis, in most gel systems, covalent-type interactions dominate, and the gel process is irreversible. The gelation process may be reversible if there are other interactions involved [2].

The purpose behind the sol-gel synthesis is to dissolve a compound in a liquid to obtain a solid controlling the factors of said synthesis. Using a controlled stoichiometry, sols of different reagents can be mixed to prepare multicomponent compounds. The sol-gel method prevents the problems with coprecipitation, which may be inhomogeneous, as it is a gelation reaction. It allows mixing at an atomic level to form small particles, which are easily sinterable.

Typically, in the sol-gel chemistry, there is a reaction of an organometallic compound, which is generally an alkoxide, nitrate, or chloride under aqueous conditions to form a solid product. This product can be a dense glass monolith, a high surface area molecular filter, an aerogel to

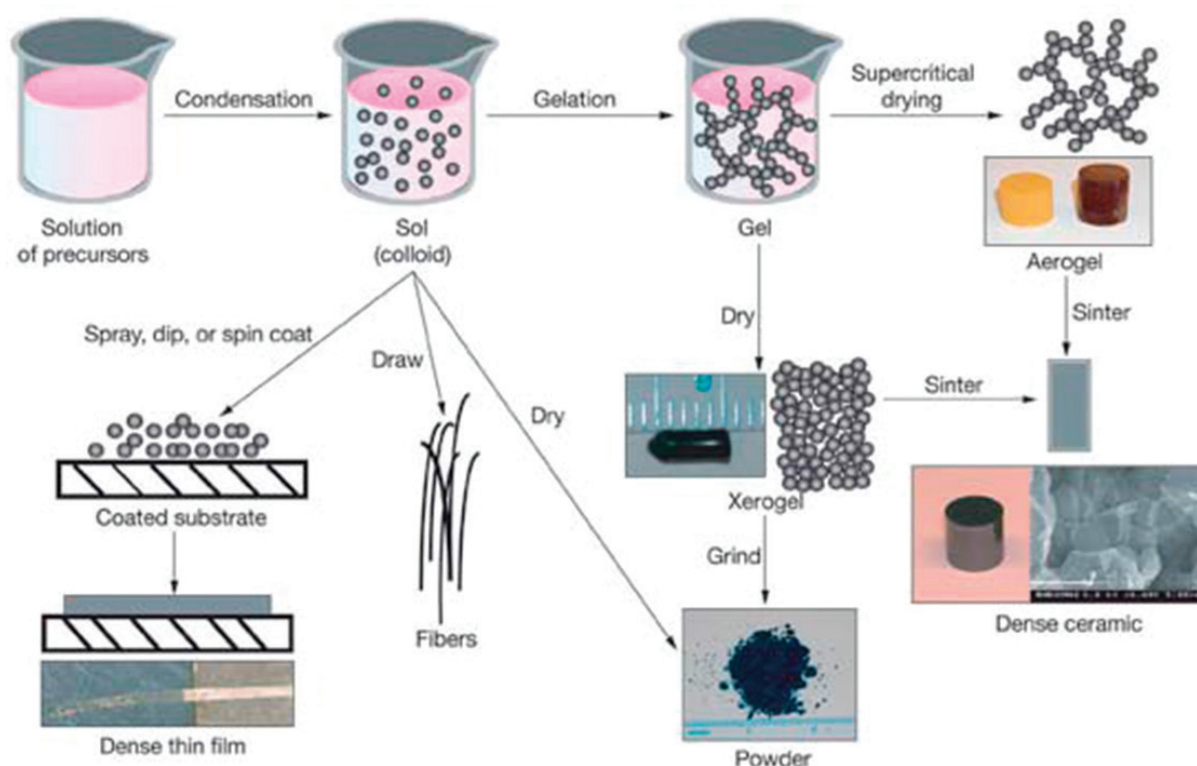


Figure 2. Stages of the sol-gel process.

a metal oxide, a nitride coating, or nanoparticle. The process begins with reactions of hydrolysis and condensation of a precursor to form a gel followed by aging, solvent extraction, and finally drying. These reactions may be catalyzed by the addition of an acid or a base, which will produce dense or diffuse networks, respectively, by altering the hydrolysis kinetics. The selection of the precursor and catalyst depends ultimately on what you would like to make [4].

In the gelation step, condensations are produced from the gel precursors in aqueous solution which are hydrolyzed and polymerized through alcohol or water. When starting the gelation, when the average size of the conglomerate is very small, they are best modeled with an approximation at the atomic level.

In the last decades, a remarkable effort has been made to develop theoretical models for this, with convincing results. In the case of hierarchically structured gels and low density gels, these cannot be analyzed directly with molecular models; a mesoscale approach should be used. In contrast, relatively dense gels can be modeled with simulations at the atomic level or coarse grain simulations. Aging of the gel is an extension of the gelation step in which the gel network is reinforced by an additional polymerization, which can be controlled by varying the temperature and the type of solvent.

In the next stage, syneresis can occur during the aging of the gel, which is the expulsion of solvent due to the contraction of the gel matrix. The process of drying the gel consists in eliminating the water from the gel system, with simultaneous collapse of the gel structure, under conditions of constant temperature, pressure, and humidity [5, 6].

Usually, the dry gel is given a calcination treatment to turn it into a crystalline material. The following reactions usually occur: desorption of solvent and water physically absorbed from the walls of micropores (100–200°C), decomposition of residual organic groups into carbon dioxide (300–500°C), collapse of small pores (400–500°C), collapse of larger pores (700–900°C), and continued polycondensation (100–700°C). The phenomena of sintering and densification are produced through different mechanisms such as condensation by evaporation, surface diffusion, grain limit, and mass diffusion.

3. Design of sol-gel materials

As mentioned earlier, the sol-gel method allows the preparation of an infinity of materials and ceramics. Its great versatility allows us to cover different areas of knowledge that cover global problems such as energy, biotechnology, electronics, health, pollution, scaffolds for tissue engineering, and smart coatings molecular imprinting [7–10]. In this way, sol-gel materials can be of different kinds, since catalysts, nanocarriers, inorganic pigments, drugs, magnetic and metallic nanoparticles. Also, it allows the encapsulation of biological molecules such as proteins and enzymes [11–13] which have applications as biosensors or the release of drugs in the treatment of neurodegenerative diseases such as cancer, Parkinson's, and Alzheimer's, for example.

Sol-gel chemistry tends to be particularly sensitive to the following parameters:

- pH: any colloidal chemistry that involves water is sensitive to pH.
- Solvent: in the polymerization process, as molecules are assembled into nanoparticles, the solvent plays two important roles; the first is that it must be able to keep the dissolved nanoparticles so that they do not precipitate out of the liquid; and second, it must play a role in helping nanoparticles connect with each other.
- Temperature: the chemical kinetics of the different reactions involved in the formation of nanoparticles and the assembly of the nanoparticles in a gel network are accelerated with temperature, which affects the gel time. At very low temperatures, gelation is a slow process that can take weeks or months. In contrast, at high temperatures, the reactions that bind the nanoparticles to the gel network occur so quickly that lumps form in their place and a solid precipitates out of the liquid. The gelation temperature must be controlled to optimize the reaction time.
- Time: depending on the type of gel to be obtained, the different steps in the gel formation process work differently at different time scales. In general, it is recommended that the formation of the gel should be slow to produce a very uniform structure, resulting in a stronger gel. Accelerating reactions through short times cause precipitates to form instead of gel network and can cause a gel to become cloudy and weak or simply not form.
- Catalysts: a chemical reaction can be accelerated by the presence of a catalyst. In much of the sol-gel chemistry, this is very pH sensitive. This is because both acids (H^+) and bases (OH^-) are catalysts but accelerate chemical reactions by different mechanisms.

- Agitation: at this stage, the mixing of the sol during gelation should ensure that the chemical reactions in the solution are produced uniformly, allowing all molecules to receive an adequate supply of the chemicals they need for these reactions to be carried out correctly. Generally, there are microscopic and macroscopic domains of gel networks partially formed throughout the liquid, and agitation can sometimes break up the formation of these domains; and the network fragments grow back into a wider network.

Therefore, taking into account these factors and the type of application, many protocols have been used to design our materials in different scales, nano-, micro-, meso-, and macromaterials, all aimed at optimizing and maximizing their optical, electrical, magnetic, and nonlinear properties [7, 8, 14]. It is described how these factors influence said properties during sol-gel reaction [7].

In this work, valuable contributions in different fields related to novel materials synthesized by the sol-gel route are shown, all with topics of great technological importance and which have an impact on engineering applications, at the level of electronics, health, and coatings.

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References

- [1] <https://www.scopus.com/search/form.uri?display=basic>
- [2] Brinker CJ, Scherer G. Sol-Gel Science. New York: Academic Press; 1989. ISBN 9780080571034
- [3] <http://solgel2019.ifmo.ru/>
- [4] Collins A. Nanotechnology Cookbook: Practical, Reliable and Jargon-Free Experimental Procedures. UK, Elsevier Science; 2012. ISBN-13: 978-0080971728
- [5] Hench LL, West JK. The sol-gel process. Chemical Reviews. 1990;**90**:33-72
- [6] Sakka S, Kozuka H. Handbook of Sol-Gel Science and Technology. 1. Sol-Gel Processing. The Netherlands: Kluwer Academic Publishers; 2004. ISBN 1-4020-7966-4
- [7] Lee BS, Lin HP, Chan JCC, Wang WC, Tsai YH, Lee YL. A novel sol-gel derived calcium silicate cement with short setting time for application in endodontic repair of perforations. International Journal of Nanomedicine. 2018;**13**:261-271

- [8] Mokhtari K, Salem SH. A novel method for the clean synthesis of nanosized cobalt based blue pigments. *RSC Advances*; **217**(7):29899
- [9] Shi C, Ding GS, Tang AN, Qiao YY. Synthesis and evaluation of ion-imprinted sol-gel material of selenite. *Analytical Methods*. 2017;**9**:1658-1664
- [10] MRD K, Shafeeyan MS, AAA R, WMAW D. Application of doped photocatalysts for organic pollutant degradation—A review. *Journal of Environmental Management*. 2017;**198**:78-94
- [11] Karataş A, Algan AH. Template synthesis of tubular nanostructures for loading biologically active molecules. *Current Topics in Medicinal Chemistry*. 2017;**17**:1555-1563
- [12] Araújo-Gomes N, Romero-Gavilán F, Sánchez-Pérez AM, Gurruchaga M, Azkargorta M, Elortza F, et al. Characterization of serum proteins attached to distinct sol-gel hybrid surfaces. *Journal of Biomedical Materials Research—Part B Applied Biomaterials*. 2018; **106**:1477-1485
- [13] Gill JK, Orsat V, Kermasha S. Screening trials for the encapsulation of laccase enzymatic extract in silica sol-gel. *Journal of Sol-Gel Science and Technology*. 2018;**85**:657-663
- [14] Ben-Arfa BAE, Miranda Salvado IM, Ferreira JMF, Pullar RC. Novel route for rapid sol-gel synthesis of hydroxyapatite, avoiding ageing and using fast drying with a 50-fold to 200-fold reduction in process time. *Materials Science and Engineering C*. 2017;**70**:796-804