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Sol-gel process for synthesis of nanoparticles and applications thereof

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Abstract
The use of sol-gel technology in leather processing processes has received huge attention in recent years. Being a chemical-oriented process sol-gel offers several benefits in medical, optical, textile, and other sectors. Particularly, it enables multifunctional properties in a single step, which is not possible due to the incompatibility of chemical materials with conventional textile finishing methods. On the other hand, the sol-gel technology has several shortcomings, including high precursor material costs. In general, metal alkoxides and metal salts have been used as precursors in sol-gel technology. In this paper, some of the extensive reports regarding the mechanism, types, processing, and steps involved in sol-gel technology were highlighted. An attempt was made to enlighten the different applications of sol-gel, especially in textiles. Rather than its advantages and disadvantages, some of the next-generation applications of sol-gel were also reported.

Keywords: sol-gel, hydrolysis, condensation, precursor, nanoparticle synthesis, surface modification, coating

1. Introduction
A sol-gel is a tedious yet simple, liquid phase synthesis technique find its use in ceramic engineering and material science. Through simultaneous adaptation, it is today widely used in the textile industry for fabrication starting from its complex solution (sol) until achieving colloidal 3D networking (gel). In the sol-gel chemical procedure, the 'sol' (or solution) evolves into a gel-like semicrystalline system containing both liquid and solid phases with morphologies ranging from discrete particles to continuous polymer networks (Uche 2013) [7]. There are various methods used to obtain narrow-sized nanoparticles and these are distinctly classified into two approaches top-down and bottom-up (Rahman and Padavettan, 2012) [11]. The top-down route is based on the bulk material and makes it smaller, thus breaking up larger particles by the use of physical processes like crushing, milling, or grinding. Typically, the pathway is not useful in preparing uniformly shaped materials, and acknowledging very small particles, even with high energy consumption, is extremely difficult. Besides ever after putting in research and costs the nanostructures may or may not retain the physical and functional properties also intended. The atom-by-atom, molecule-by-molecule, or cluster-by-cluster construction of a material is referred to as a bottom-up approach. Most nano-scale materials with the ability to generate a uniform size, shape, and distribution are prepared using this method. Chemical synthesis is effectively covered, and the reaction is precisely controlled to prevent further particle growth. In the modern era, where green technology is an emerging trend in textile manufacturing and finishing, competing to that sol-gel technology has also evolved in the same area owing to its less chemical use, low cost, more effective, functionality, and durability with low environmental impact and toxicity. Sol-gel is a purely wet processing, chemical-based process that entails the formation of inorganic networks by establishing a colloidal suspension (sol) and gelating it to form a network in a continuous liquid phase (gel) (Ismail, 2016) [6].
Hereby, this paper enlightens the suitable mechanism and related application of the sol-gel method in the textile industry.

2. Mechanism
In the preparation of the "sol", the initial products are mostly inorganic metal salts such as metal alkoxides also called precursors. In a standard sol-gel phase, the precursor is subjected to a series of hydrolysis and polymerization reactions to form a colloidal suspension, or "gel" (Sajjadi 2005).

The detailed mechanism included in the sol-gel process is enlightened below (Uche 2013) [7].

Si $(OC_2H_5)_4$, or Si $(OR)_4$ is the chemical formula TEOS (tetraethyl orthosilicate) is a precursor of silicon oxide, where, C_2H_5 is the alkyl group.

Si $(OR)_4 + H_2O \rightarrow$ HO-Si $(OR)_3$ + R-OH ---Hydrolysis
OR groups were replaced by OH groups
Si $(OR)_4 + 4H_2O \rightarrow$ Si $(OH)_4$ + 4R- OH
$(OR)_3$–Si-OH + HO–Si-$(OR)_3 \rightarrow$ [$(OR)_3$Si–O–Si $(OR)_3$] + H-O-H --Polycondensation
OR
$(OR)_3$–Si-OR + HO–Si-$(OR)_3 \rightarrow$ [$(OR)_3$Si–O–Si $(OR)_3$] + R-OH

Polymers are the huge macromolecules formed with addition or hundreds of monomers. Because the fully hydrolyzed monomer Si$(OH)_4$ is tetrafunctional, polymerization of silicon alkoxide can result in complex branching of the polymer (can branch or bond in 4 different directions). As a result, polycondensation is linked to the rapid formation of three-dimensional networking of siloxane bonds [Si–O–Si], as well as the production of H-O-H and R-O-H species, as the liberation of water or alcohol (Jung *et al*. 2000) [7]. In general, several stages are identified in this process, beginning with a silicate solution and progressing to the formation of a sol, which will then be transformed into a gel, and finally, the formation of a dry gel, which is generally formed by a three-dimensional network of silica with numerous pores of various sizes interconnected. The routes of this mechanism are depicted in Figure 1.

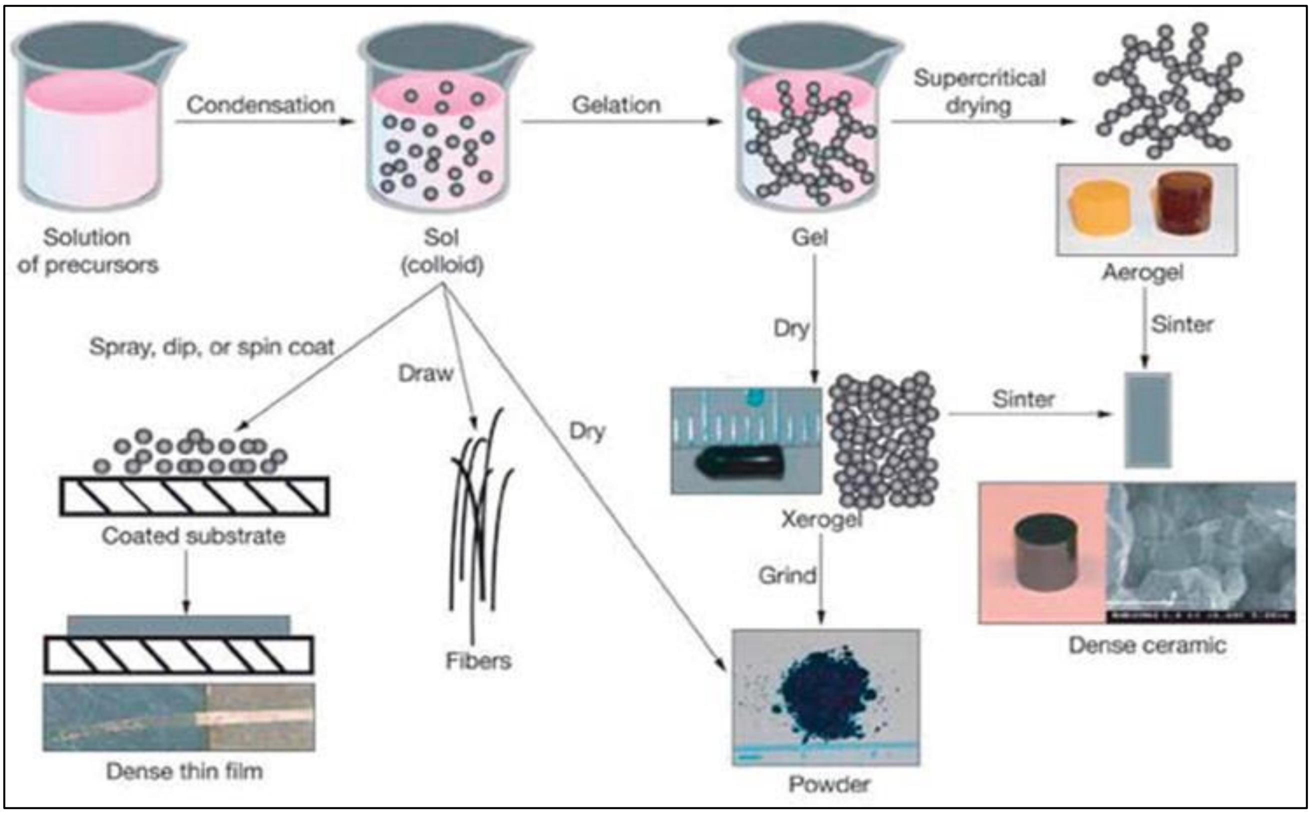

Fig 1: Stages of the sol-gel process (Aguilar 2018)

3. Various steps involved in sol-gel process

The steps involved in sol–gel processing are as follows (Ullattil and Periyat 2017) [18]

a. Preparation of a homogeneous liquid precursor solution in an organic solvent that is miscible with water or other common reagents.
b. Treatment of a homogeneous solution with a suitable reagent to convert it to a sol. Here, molecular manipulations such as adjusting the hydrolysis conditions to achieve precisely controlled particle sizes, adding dopants to achieve special properties in the desired product, and so on are possible.
c. Self-polymerization and condensation are used to turn the sol into a gel. This sol–gel conversion has a wide range of applications in the industrial and catalysis fields.
d. Creating desired shapes from the gel, such as thin films, fibres, spheres, and so on.
e. Sintering the gel into the desired shape in ceramic materials.

3.1 The details of the so-gel process depicted in figure 2

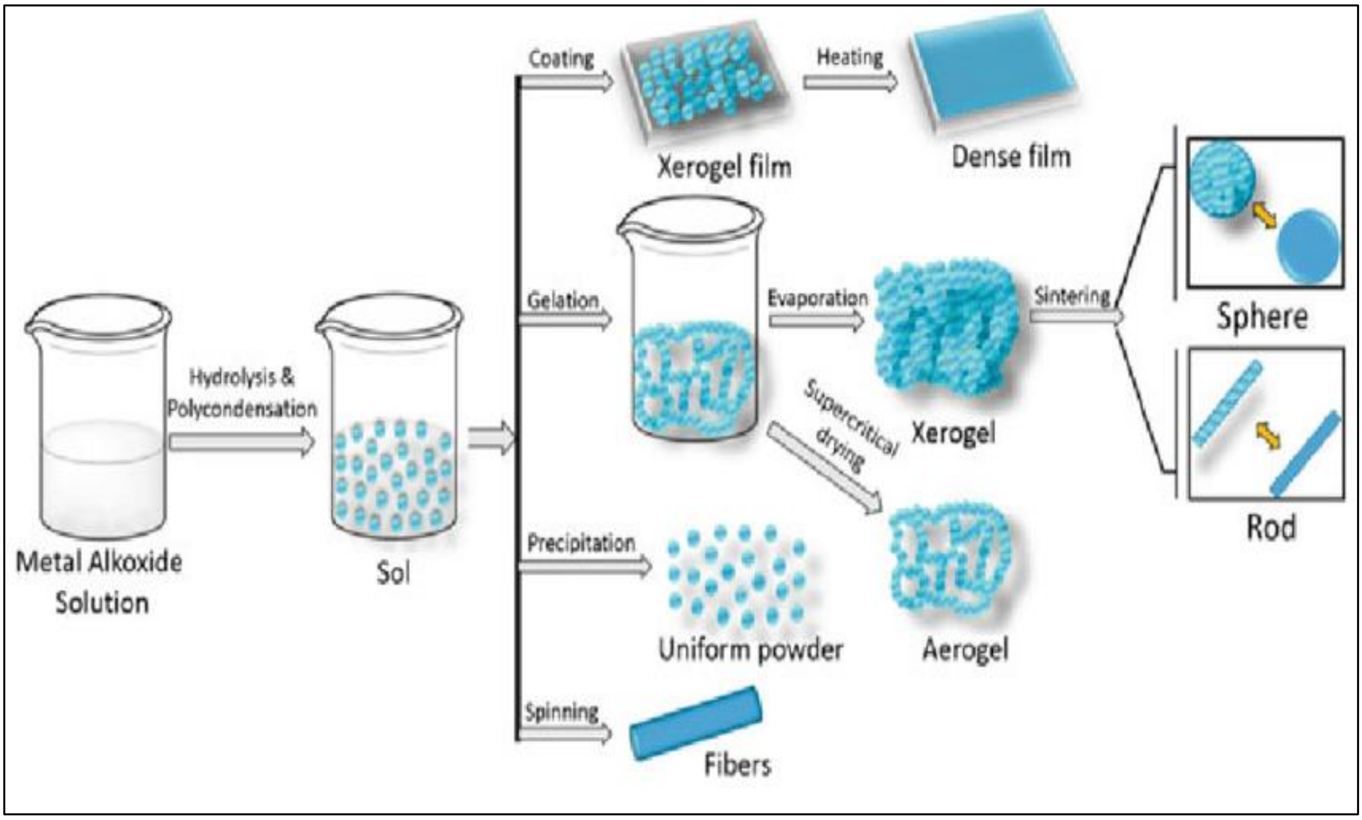

Fig 2: Schematic representation of sol–gel processing (Ullattil and Periyat 2017) [18]

4. Factors affect the sol-gel method

The following parameters are especially important in sol-gel chemistry (Aguilar 2018) [1]:

- **pH:** pH is important in any colloidal chemistry that involves water.
- **Solvent:** As molecules are assembled into nanoparticles during the polymerization process, the solvent plays two important roles: first, it must be able to keep dissolved nanoparticles from precipitating out of the liquid, and second, it must assist nanoparticles in connecting with one another.
- **Temperature:** It accelerates the chemical kinetics of the various reactions involved in the formation of nanoparticles and the assembly of nanoparticles in a gel network, affecting the gel time. Gelation is a slow process that can take weeks or months at very low temperatures. The reactions that bind the nanoparticles to the gel network, on the other hand, occur so quickly at high temperatures that lumps form in their place and a solid precipitate out of the liquid. To optimise the reaction time, the gelation temperature must be controlled.
- **Time:** The different steps in the gel formation process work differently at different time scales depending on the type of gel to be obtained. In general, it is recommended that the gel be formed slowly in order to achieve a very uniform structure and a stronger gel. Accelerating reactions for short periods of time causes precipitates to form instead of a gel network, resulting in a cloudy, weak, or non-forming gel.
- **Catalysts:** The presence of a catalyst can speed up a chemical reaction. This is very pH sensitive in much of the sol-gel chemistry. This is because both acids (H^+) and bases (OH^-) are catalysts, but they work in different ways to speed up chemical reactions.
- **Agitation:** At this point, the mixing of the sol during gelation should ensure that the chemical reactions in the solution are uniformly produced, allowing all molecules to receive an adequate supply of the chemicals they require to carry out these reactions correctly. In general, microscopic and macroscopic domains of gel networks are partially formed throughout the liquid, and agitation can break up the formation of these domains, allowing the network fragments to grow back into a larger network.

5. Application of sol-gel in textile production

Sol-gel technology has been studied in the textile industry since 1960 and is well known throughout the materials, metallurgy, ceramics, and glass industries. A couple of applications in the textile finishing process include nanoparticle synthesis, dip and thin coatings for flame retardant, ultraviolet (UV) protection, water/oil repellent, antibacterial, self-cleaning, and anti-wrinkle protection. In addition, the sol-gel technique has been shown to significantly improve the surface behavior of the fabrics after altering the surface topography. A schematic diagram Figure 3, represents the application and versatility of sol-gel technique.

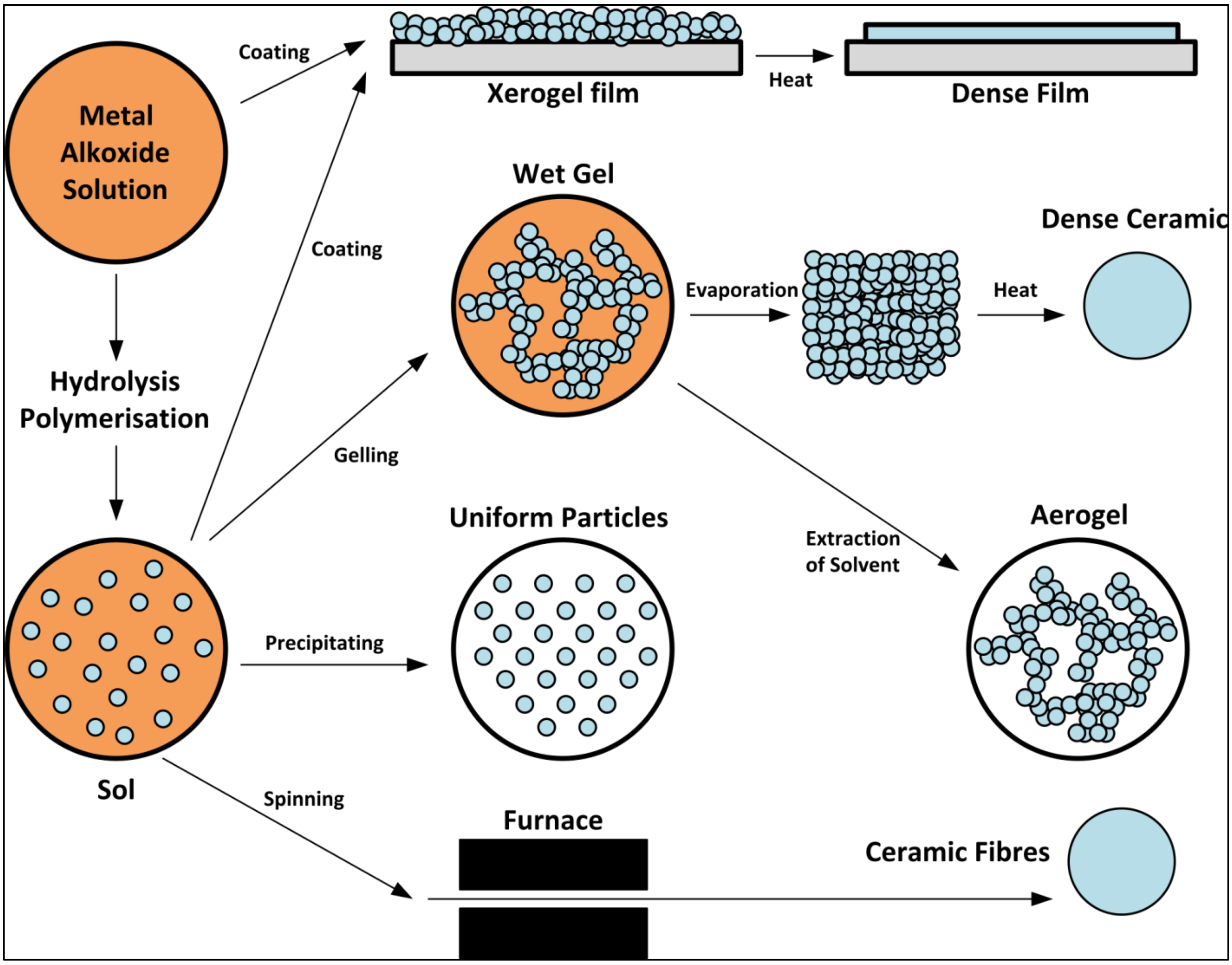

Fig 3: Schematic of sol-gel application (Trindade and Politi 2019) [16]

5.1 Synthesis of nanoparticles for textile finishing

Sol-gel method is very well known for nanoparticle synthesis, as it is associated to the chemical wet synthesis method, it is possible to achieve very narrow sized NPs with optimized conditions. Commonly, the sol-gel based precipitates are amorphous in nature, therefore further heat treatment is given to induce required crystallization (Chellappa and Vijayalakshmi 2017) [4]. An annealing temperature higher than 300 °C is generally needed to induce the transition from the amorphous to the anatase stage, resulting in dramatic particle size growth (Selvaggi *et al*. 2015) [14]. In this regard, Kartini *et al*. (2004) [9] reported the two-step sol-gel synthesis of mesoporous titanium NPs. The hydrolysis of titanium isopropoxide in a specific aqueous solution induced by a neutral surfactant was the first step in the development process.

5.2 Nano-dyeing

Sol-gel derived dyeing incorporates the dye molecules inside the gelatinous 3D structure and acts as a barrier protecting them from photodegradation. This improves the durability of the dye and the colour fastness properties of dyed fabric. Furthermore, if the dye molecule is acquired by a metal complex, enhancement in other functional properties like UV absorbent, photocatalytic, self-cleaning, *etc*, were observed. Nevertheless, these functional properties are solely depending upon the synthesized metal particles through sol-gel. In order to achieve functionality and colour Kale *et al*. (2016) [8] dyed cotton and its blended polyester fabrics with basic dye, acquiring sol-gel technique. Successive colorfastness was achieved with this combined process and owing to the presence of silica sols water repellent property is also achieved.

5.3 Fiber extraction

Nanofibres have smaller pores and a larger surface area than normal fibers, have wide applications in nano-catalysis, tissue scaffolding, protective clothing, filtration, and optical electronics. The electrospinning method uses a high voltage electrical field to create electrically charged jets of polymer solution or melts formed by nanofibers on drying by evaporation of the solvent (Subbiah *et al*. 2005) [15]. Franco *et al*. (2018) [5] developed polymeric nanofibers from nylon polymer (PA 6/66) with an optimized sol-gel based electro spinning technique. With aiding acetic acid and formic acid as solvents 3 coagulant solution baths of different concentrations i.e., 12% wt., 17% wt., and 22% wt., were prepared with continuous stirring. The average diameter of the obtained nanofibers rages between 117 to 660 nm.

5.4 Dip and thin coating

Dip coating is the modest, cost-effective, relevant, and reproducible process involving the adsorption of wet liquid film by immersing the substrate in a solution containing compounds of hydrolyzable metal (or readily formed particles) and its withdrawal into an atmosphere containing water vapor at a constant pace (Brinker *et al*. 1991; Brinker and Hurd 1994; Lu *et al*. 1997) [3, 2, 10]. The sol-gel method, in which thin films are formed by dipping and polymeric or particulate inorganic precursors is concentrated on the surface of the substrate used for dip coating through a complex process involving gravitational draining, concurrent drying, and sustained condensation reactions. Structured films with thicknesses ranging from monolayers (nanometer) to several micrometers were deposited by polymeric precursors, depending on factors such as precursor size and structure, relative rates of condensation and evaporation, capillary pressure, and substrate withdrawal speed (Scriven 1988) [13]. Thin films are commonly thought of in connection with semiconductors. However, thin films are important in a multiplicity of other areas where coatings of only a few microns thickness are needed. The thin film is valuable since it alters the surface interactions of the newly formed platform by separating the bulk substrate properties from the surface interactions.

6. Conclusion

From the beginning stages of processing, the sol-gel technique aims to control the material's dimensions on a nanometer scale. Chemical processing, controlled high purity, and improved homogeneity can all help improve material properties. This low-temperature processing method has a number of advantages over traditional nanoparticle synthesis methods. Film coating and fiber pulling are two other advantages reported reciprocally. Sol-gel technology is dedicated to the ability to modify material properties and combine multiple functionalities in a single material. The use of low temperature conditions and insensitivity to the environment are two of the most ensured adequate of sol-gel technology. These features enable a range of materials to be used, including textile substrates which do not tolerate high temperatures. Innovative UV-protected fabric finishing, antibacterial, flame retardant, water repellent, self-cleaning properties, or a combination of properties are now possible due to the combination of innovative sol-gel materials with specific functionality.

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