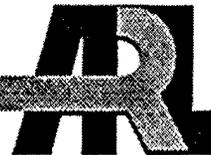


ARMY RESEARCH LABORATORY



Overview of Sol-Gel Science and Technology

by Sandra K. Young

ARL-TR-2650

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Overview of Sol-Gel Science and Technology

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Abstract

Sol-gel science and technology continues to interest researchers decades after its discovery. The reaction is easy to perform, does not require special conditions (can be done on the bench top in a beaker), and does not require high temperatures. The sol-gel reaction consists of a series of simple hydrolysis and condensation reactions. This reaction can be influenced by several factors: quantity of acid catalyst, amount of water used, incorporation of a network modifier, and varying solvent effects. This work presents an overview of sol-gel science and technology, discusses the advantages of sol-gel reactions, and describes the factors that can influence changes in the final sol-gel material.

Acknowledgments

The author would like to thank Dr. Nora Beck Tan and Dr. Steven McKnight, who encouraged and continue to encourage independent scientific pursuits in the polymers field, which led to some in the sol-gel field, and acknowledge the support of the polymers branch in continuing research in the area of sol-gel science and technology.

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1. DOD Interest in Sol-Gel Science and Technology

Along with the demand for new technologies, new processing techniques, and novel nanomaterials, there has been an increased interest in investigating sol-gel science and technology. The Department of Defense (DOD) community is looking at sol-gel science to produce high-performance materials for low gravity and high impact, possessing properties such as increased toughness, strength, and thermal stabilities, and decreased sensitivity to shock. These materials encompass applications from membranes for fuel cells or water purification, to composites for space vehicles or planes, to encapsulated nanomaterials for energetics. Researchers are finding that sol-gel science is a straightforward technique that many scientists have been using to produce inorganic and organic-inorganic materials.

2. Global Overview of Silicon-Based Research

Silicon is the second most abundant element on earth, exceeded only by oxygen [1]. Silicon-containing compounds, in a variety of compositions, account for over 90% of the earth's land mass [1, 2]. The earth's crust alone consists of 25.7% silicon, by weight. Silicon is not found free in nature, but occurs chiefly as an oxide and as silicates [1]. Sand, quartz, rock crystal, amethyst, agate, flint, jasper, and opal are some of the forms in which the oxide appears [1]. It is through this natural abundance of silica as a source of raw materials, in addition to its inherent high thermal stability and high mechanical strength, that has drawn increased emphasis on practical applications of silicon-based materials in the research and development of high-performance and functional materials.

Silicon-based research has drawn much attention in recent years, with targeted conferences such as the University Pierre et Marie Curie hosting proceedings on hybrid organic-inorganic materials [3], the National American Chemical Society hosting symposia on silicones and silicone-modified materials [4], and McMaster University organizing an annual organosilicon symposium [5]. In addition, publications such as the *Journal of Sol-Gel Science and Technology*, *Chemistry of Materials*, and the *Journal of Non-Crystalline Solids* contain a significant number of articles that focus on the research of silicon compounds. These conferences and journals highlight much of the major silicon-based research generated in academia and industry. Research involving reactive silicone chemistry includes production of pure silicon to semiorganics, ring-opening and atom transfer polymerizations, hydrosilylation, polymerizations with controlled

stereochemistry, and condensation reactions. These reactions with silicones produce a wide range of materials which encompass the major areas of silicon-based research and chemistry: polymers, elastomers, interpenetrating networks, reinforcing fillers, membranes, microlithography, photoinitiation, high-performance polymers, and sol-gel-derived ceramic precursors.

3. Introduction to Sol-Gel Science and Technology

While it was studied earlier, sol-gel chemistry has been investigated extensively since the mid-1970s, when sol-gel reactions were shown to produce a variety of inorganic networks that can be formed from metal alkoxide solutions [6]. Through sol-gel processing, homogeneous, high-purity inorganic oxide glasses can be made at ambient temperatures rather than at the very high temperatures required for conventional formation of glasses. Various products, such as molded gels [7, 8], spun fibers [9, 10], thin films [11-13], molecular cages [14, 15], and xerogels [16] have been developed for utility in such areas as gas separations [12, 17], elastomers [18, 19], coatings [20, 21], and laminates [17, 22]. It is through the inorganic compound incorporation into organic polymers that a wide variety of property modifications can be achieved (see Figure 1).

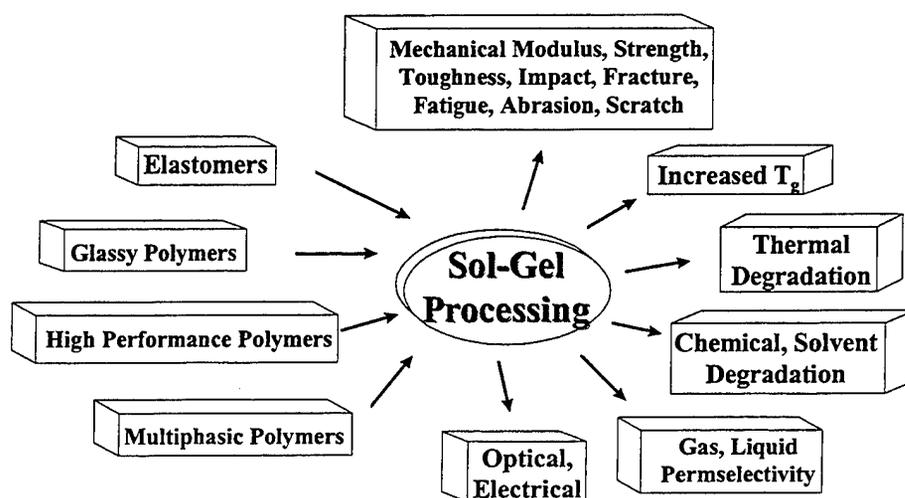


Figure 1. Potential applications for polymer material modification utilizing *in situ* sol-gel processes.

The sol-gel chemical process is self-described in the definition of a sol, a gel, and a summary of the process in which a sol evolves into a gel [23] (see Figure 2). A sol is a colloidal dispersion of small particles in a liquid; a gel is usually a

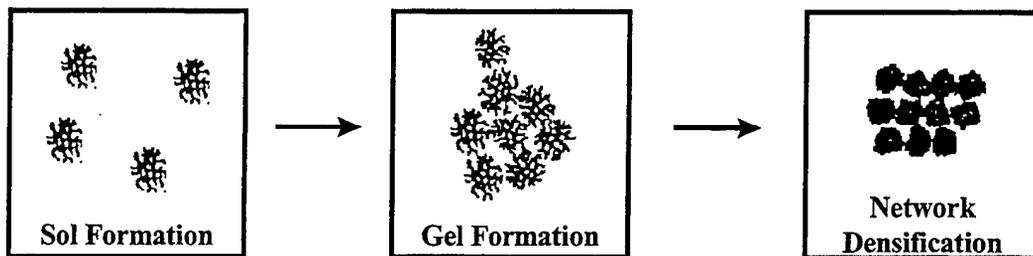


Figure 2. Colloidal network formation in sol-gel materials.

substance composed of a continuous network encompassing a continuous liquid phase [24, 25]. Sol-gel reactions promote the growth of colloidal particles (sol) and their subsequent network formation (gel) through the hydrolysis and condensation reactions of inorganic alkoxide monomers. The precursors for synthesizing these colloids consist of a metal or metalloid element surrounded by various reactive ligands. Metal alkoxides are most popular because they react readily with water. The most widely used metal alkoxides are the alkoxysilanes, such as tetramethoxysilane (TMOS) and tetraethoxysilane (TEOS). However, other alkoxides such as aluminates, titanates, zirconates, and borates are also commonly used in the sol-gel process, either alone or in combination with other alkoxides such as TEOS.

To more fully understand the sol-gel chemical process, the sol-gel reaction must be examined. Sol-gel reactions are a series of hydrolysis and condensation reactions of an alkoxysilane, which proceed according to the reaction scheme shown in Figure 3. Hydrolysis is initiated by the addition of water to the TEOS solution under acidic, neutral, or basic conditions.

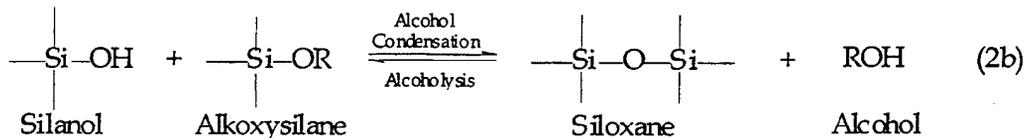
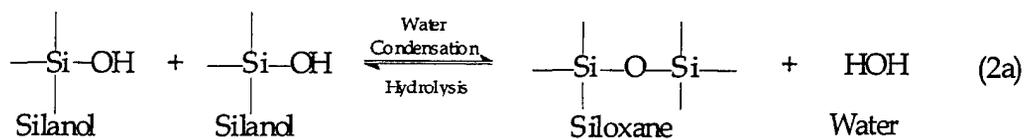
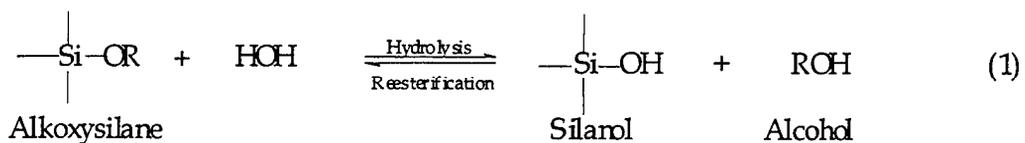


Figure 3. Sol-gel general reaction scheme.

In the first step, hydrolysis, a silanol group is generated (Figure 3, reaction [1]). As only acid catalysis is used in this research, discussions of the sol-gel reactions will refer to acid catalysis only [25]. A positive charge develops on the alkoxy silane through the attack of an acid catalyst. The alkoxy silane then is hydrolyzed in an Sn2-type reaction, forming a silanol moiety. This is discussed more thoroughly in section 3.3, which deals with type and amount of catalyst. The mechanism for reaction 1 is depicted in Figure 4. TEOS is not soluble in water. Therefore, hydrolysis is promoted by the addition of organic cosolvents, such as alcohols [25] that aid in the thorough mixing of the alkoxide molecules with water molecules in solution.

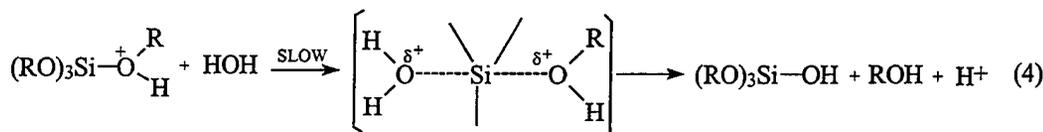
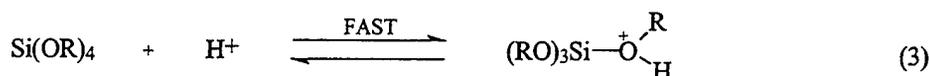


Figure 4. Hydrolysis mechanism for a tetraalkoxysilane.

In the second step, the silanol group can undergo condensation with either an alkoxide or another silanol group (reaction [2a] or [2b] in Figure 3), which results in the formation of strong siloxane linkages and produces either alcohol (ROH) or water *in situ*. As the number of Si-O-Si bridges increases, the siloxane particles can aggregate into a sol. This, as shown in Figure 2, consists of the formation of small silicate clusters dispersed in solution. Gel formation occurs when the sol particles undergo sufficient condensation reactions such that a network (a gel) is formed, trapping the aqueous and alcohol by-products. After gel-network formation is complete, the by-products are removed by heat and vacuum, yielding a vitrified and densified glass network.

3.1 Factors Influencing the Sol-Gel Reaction

A number of conditions can influence the hydrolysis and condensation reactions. Of these, the most pertinent include water-to-alkoxide ratio, type and amount of catalyst, type of organic group(s) attached to the silicon atom, and solvent effects [25, 26]. These factors will now be considered.

3.2 Water-to-Alkoxide Ratio

The effect of the water-to-alkoxide ratio for the sol-gel process is such that as the ratio increases, so does the SiO₂ content of the gel. Therefore, for complete hydrolysis, there must be at least one mole of water for every alkoxide group. Some researchers have gone further to state that if more than one mole of water

per alkoxide group is used, the reverse reaction, reesterification, will occur faster than the forward reaction [25]. However, in a recent article by McCormick et al. [27], whose experiments were conducted over a wide range of water-to-TEOS ratios, there was no correlation between the water:alkoxide ratio and the achievement of complete hydrolysis. The validity of these experiments on the effect of water:alkoxide ratio are thought to be accurate because water is generated in the reaction *in situ* and therefore the reaction, once catalyzed, self-propagates the hydrolysis.

3.3 Type and Amount of Catalyst

One important issue for consideration when deciding the concentration of catalyst is whether a precise concentration is needed. In the sol-gel process, water is generated *in situ* through condensation reactions. This makes calculation and addition of any precise amount of catalyst difficult. In addition to water:TEOS ratio experiments, McCormick et al. [27] synthesized sol-gel films, adding a wide range of acid concentrations. Their results indicated no correlation between the acid concentration and acid initiation of the sol-gel reaction. In the experiments, different acid concentrations were used with each sample and, as previously discussed, the condensation reaction caused the *in situ* generation of water. Consequently, this *in situ* generation of water diluted the initial acid concentration in every experiment. However, no differentiation between the final sol-gel structures was observed by ^{29}Si nuclear magnetic resonance (NMR) spectroscopy for the various different overall sol-gel reactions. From these experiments, it was determined that only a catalytic amount of acid was necessary. Therefore, the reaction, containing this minimum amount of catalyst in all experiments, could self-propagate. This may cause a change in the kinetics of the reaction but not in the basic structure of the overall network.

Hydrolysis and condensation reactions of most inorganic alkoxides can be carried out without catalyst because of the extremely fast rates of reaction. However, alkoxysilanes hydrolyze much more slowly, requiring the addition of either an acid or base catalyst (see Figure 5). Acid-catalyzed reactions, having a particle nucleation rate-determining process, tend to yield more linear-like networks due to the fast hydrolysis. Therefore, acid catalyzed systems have a less completely formed network of siloxane bonds with a higher concentration of unreacted silanols. Base-catalyzed reactions, on the other hand, yield highly dense materials due to the longer time that the sol particles have to aggregate and arrange themselves in the most thermodynamically stable arrangement. This process leaves fewer unreacted silanol groups in the overall network and a more highly densified network.

Acid catalysis, used in this research, increases the rate of the hydrolysis reaction, as shown in Figure 4. In the first step (Figure 4, equation [3]), hydrolysis occurs

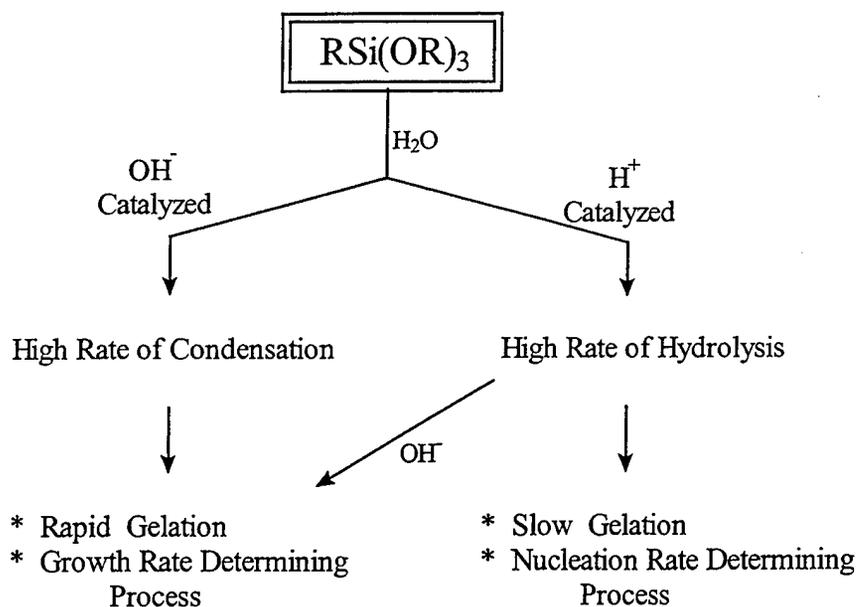


Figure 5. Effect of catalyst on hydrolysis and condensation.

through the protonation of the oxygen atom of the alkoxy group due to the presence of extra electrons. This causes a shift in the electron cloud of the Si-O bond toward the oxygen, resulting in a positive charge on the silicon atom [28]. In the rate-determining step (Figure 4, equation [4]), oxygen from the water attacks the silicon atom, which has low electron density. This results in the formation of a pentacoordinate transition state in which partial positive charges are developed. Steric effects, such as the presence of bulky and/or long alkyl substituents, will affect the rate of the hydrolysis reaction by hindering the inversion of the S_n2 transition state [28].

Several pH-dependent rate profiles have also been reported for the hydrolysis and condensation reactions, where it has been found that reaction rates are largely dependent on pH (see Figure 6) [24, 25, 29, 30]. For instance, at $\text{pH} \approx 7$, molecular hydrolysis occurs at a slow rate, while molecular condensation occurs at a fast rate. It is this inverse relationship between the rates of the hydrolysis and condensation reactions that must be taken into account in controlling the kinetics of the reaction and therefore controlling the ultimate network structure.

3.4 Type of Network Modifier

When TEOS is commixed with a silicon alkoxide comonomer containing organic groups directly bonded to the silicon atom, organically modified silicates (ORMOSILs) are created. The result of synthesizing ORMOSILs is to modify the network connectivity. Organic groups cause coordination centers with

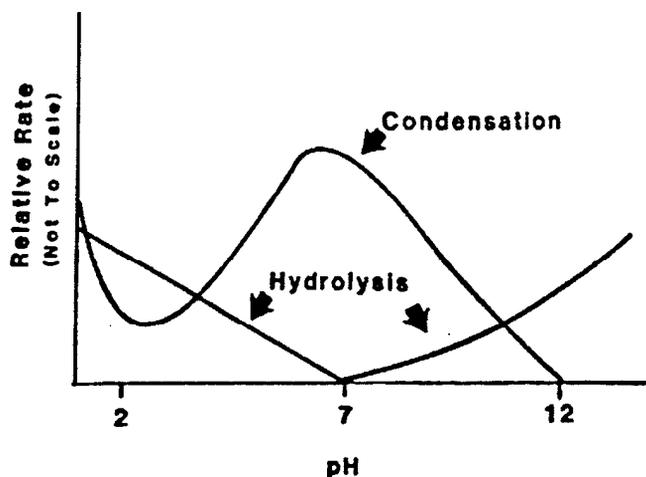


Figure 6. Effect of pH on hydrolysis and condensation rates.

functionality less than four and influence the reactivity of the alkoxy groups and therefore the connectivity of the sol-gel network in two ways. (1) Formation of siloxane bonds requires the diffusion of partially hydrolyzed molecules. The larger the alkyl group attached to the silicone, the slower the rate of diffusion and therefore the less interconnection within the network. (2) It is also reported that larger alkyl groups produce polymers with higher surface area [25]. Larger surface area allows for higher unreacted oxide concentration in the gels. This produces a branching effect in the sol-gel network. In addition to these two points, it was previously discussed that steric effects, such as the presence of bulky and/or long alkyl substituents, will affect the rate of the hydrolysis reaction by hindering the inversion of the S_N2 transition state [28]. ORMOSILs will be discussed in more detail in a subsequent technical report on organic-inorganic nanocomposites.

3.5 Solvent Effects

The effect of solvents on hydrolysis and condensation reactions is not usually discussed primarily because solvents other than water and simple alcohols, which are produced internally, are not often used. However, addition of external solvents can have an important effect on controlling hydrolysis and condensation rates. These solvents, excluding the usual water and alcohol cosolvent, are called drying chemical control additives (DCCAs) and include tetrahydrofuran, formamide, dimethylformamide, and oxalic acid. These solvents can be used in addition to alcohol in order to promote slow drying of the silica monolithic gel [25, 28]. In bulk films, slow drying is necessary to prevent cracking of the film.

Condensation rates may be dramatically affected by solvent type (see Figure 7). Condensation, via acid catalysis, is proposed to go through a two-step S_N2 mechanism.

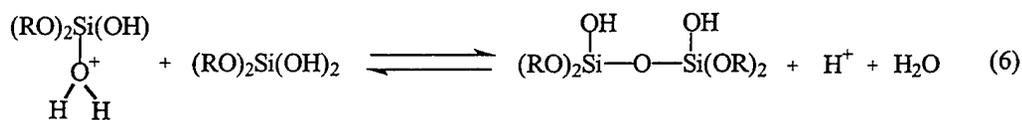
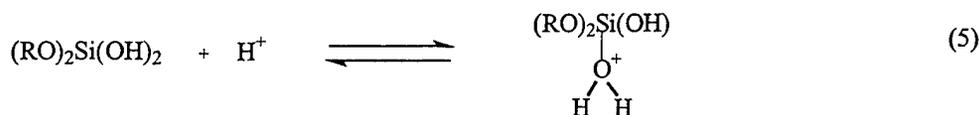


Figure 7. Condensation mechanism for a tetrafunctional silicate.

Protic solvents seemingly have little or no effect on condensation rates. Polar aprotic solvents, such as those previously mentioned, solvate the protonated silanol ($Si-OH_2^+$). The cation is solvated by orienting the negative end of the polar aprotic around the cation and by donating unshared electron pairs to the vacant orbitals of the cation. This process stabilizes the transition state and increases the rate of condensation. Use of DCCAs can be an advantage for sol-gel reactions within a membrane template. A DCCA will allow molecular diffusion but will delay the reaction, resulting in inorganic uniformity through the template. Without a DCCA, the overall reaction rate is high in relation to the slow diffusion process; therefore, the ultimate geometrical distribution of the silicate phase will be highly nonuniform.

4. Summary

Sol-gel science and technology has the potential to make a significant impact in modification of the properties of materials. One of the most significant benefits of sol-gel science is its use of room temperature conditions and insensitivity to the atmosphere. These features will allow its use with various materials, which cannot tolerate high temperatures and does not limit the researcher to using special precautions, such as a dry box. By assuming the load- and thermal-bearing phase in the material, sol-gel materials can increase thermal and mechanical properties of composites if the two systems are mixed homogeneously on the molecular level. A number of factors, including water content, modifiers, and solvent, can be altered in any sol-gel system, modifying the connectivity of the network and affecting the properties.

Several current projects in the polymers branch utilize sol-gel chemistry. These include silane epoxies for adhesives, silane urethanes for controlled porosity materials for water filtration, silane zeolites for modifying polymer membranes for fuel cells or chemical/biological resistant materials, and silsesquioxane materials for compatibilizing organic-inorganic composites. It is likely that technical reports and peer-reviewed publications will come out of these works in the near future.

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