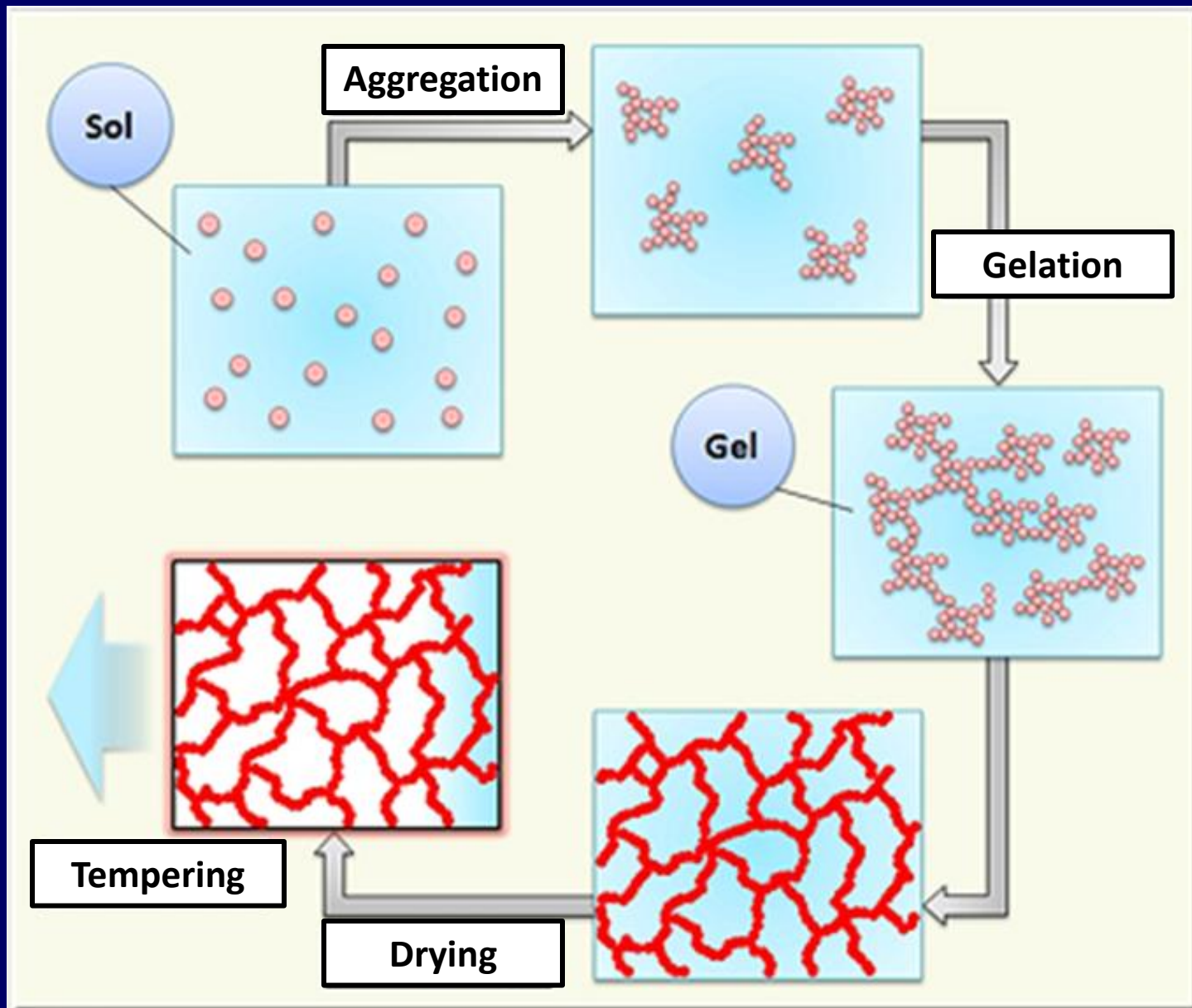
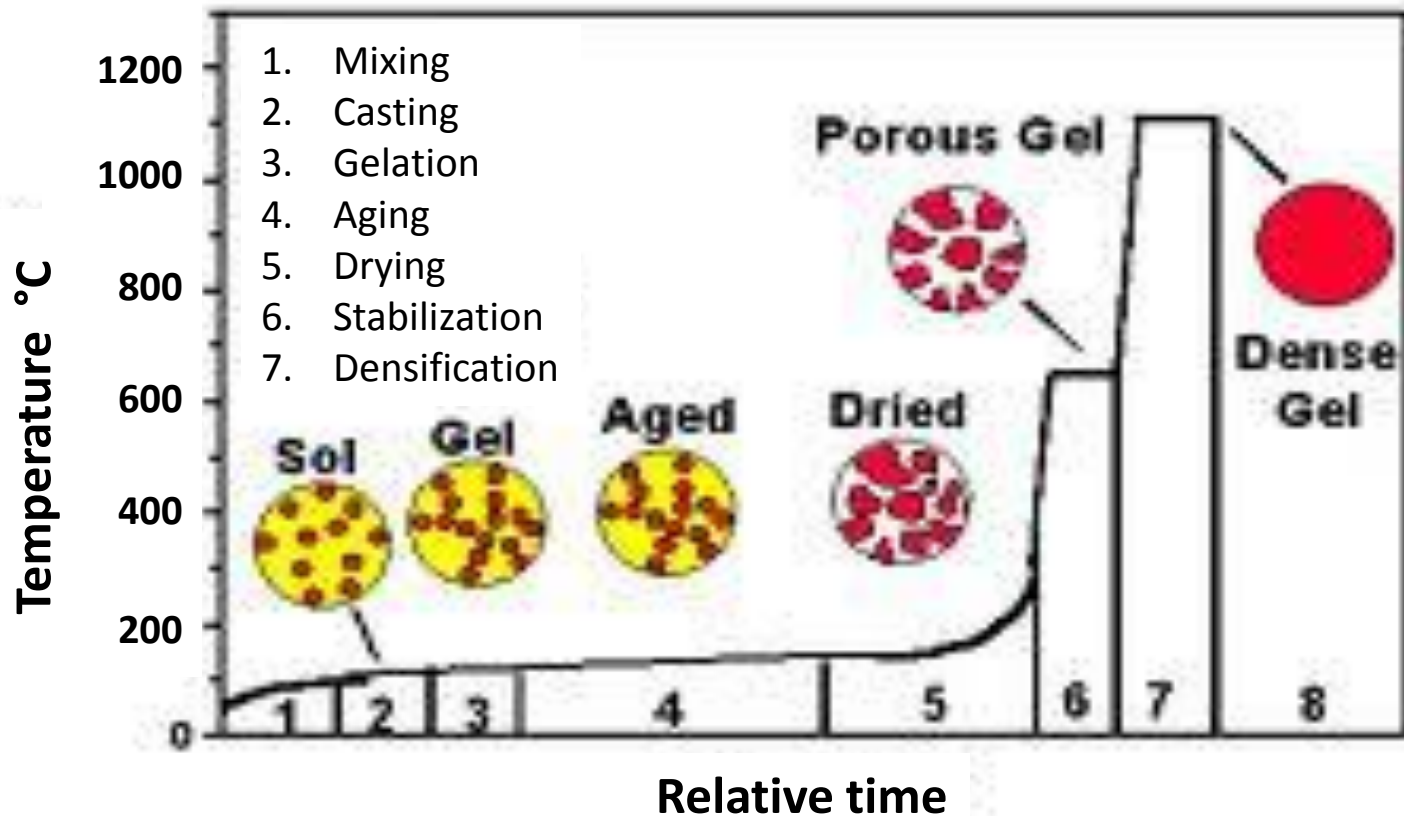


Introduction to Sol-Gel



Sol-Gel Process Steps

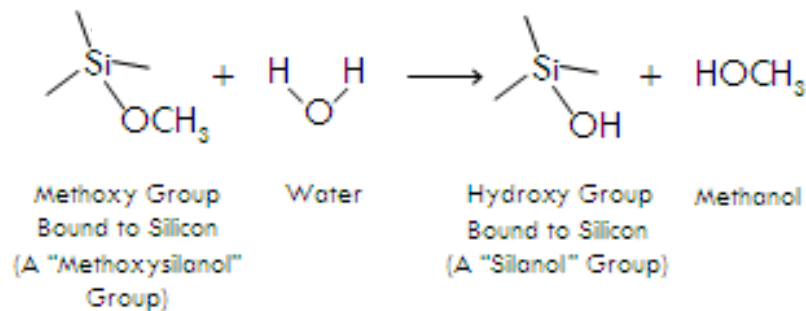


Gel Glass Process Sequence

Sol-Gel Process: The Chemistry

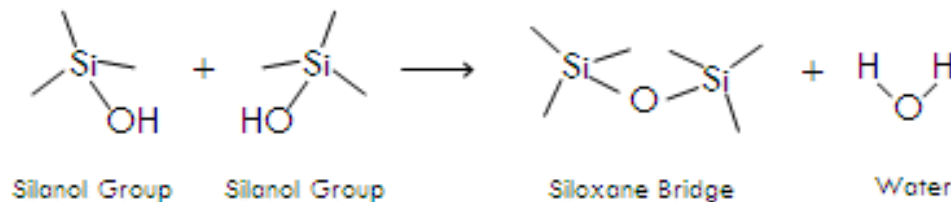
The Three Primary Reactions in Silica Gel Formation via the Alkoxide Technique

Hydrolysis



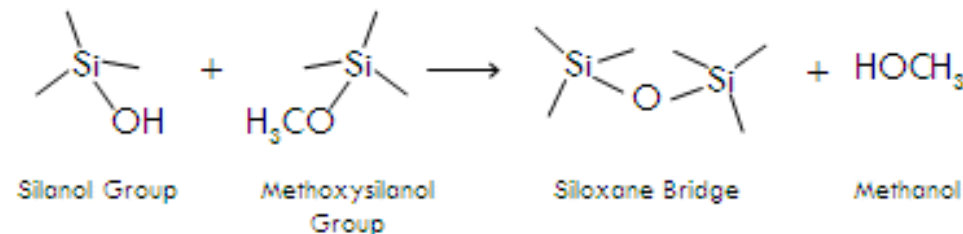
This reaction produces the reactive groups which will form polymer links

Water Condensation



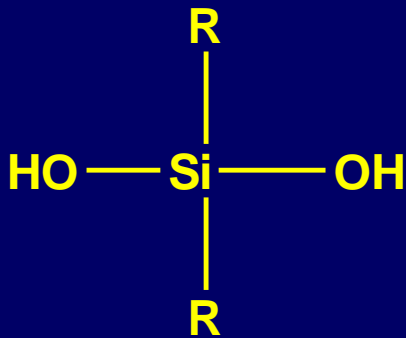
Once silanol groups form, they can condense through two different reactions to form connective silicon-oxygen-silicon bridges

Alcohol Condensation

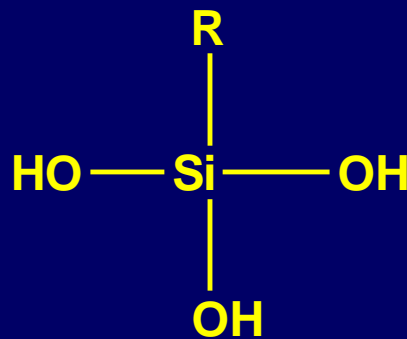


Functionality Determines Properties

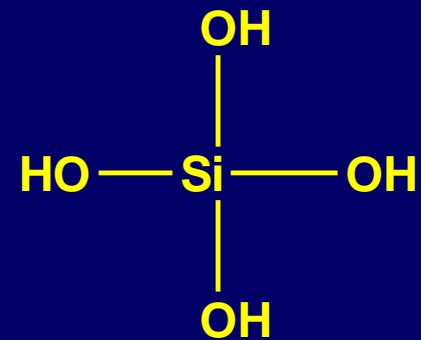
- Bifunctional – produces linear or ring structures
- Trifunctional – produces crosslinking
- Tetrafunctional – produces crosslinking



bifunctional



trifunctional

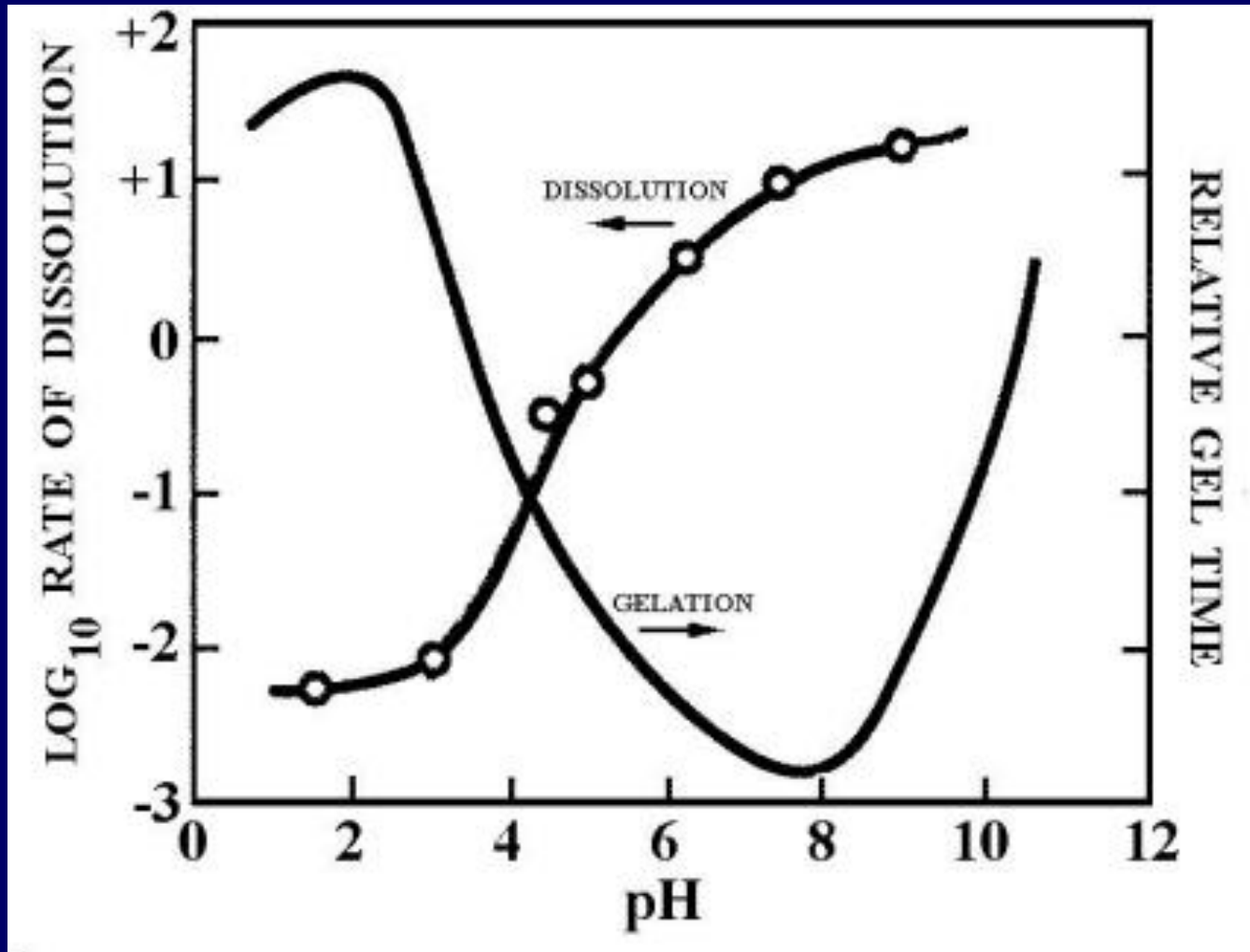


tetrafunctional

Factors Affecting Chemistry

- $\text{H}_2\text{O}/\text{Si}$ Molar Ratios
 - Temperature and Time
 - pH
 - Catalyst
-
- It is possible to vary the structure and properties of the network by controlling these factors.

pH Affects Chemistry



Sol-Gel Process: Gelation

- When the nanoparticles reach a critical size they stop growing and begin to agglomerate with other nanoparticles.
- When enough of the nanoparticles join together that a continuous network spans the liquid solution, a gel has formed.



Sol-Gel Process: Gelation

- Gelation Time – more viscous system; time dependent on pH, temperature, R groups etc.
- Viscosity of the System – related to size of particles, pH, solvent, etc

- **Acid-catalyzed**

- yield primarily linear or randomly branched polymer



- **Base-catalyzed**

- yield highly branched clusters



Sol-Gel Process: Aging

- Polycondensation – reactions continue to increase network
- Syneresis – spontaneous shrinkage; continues until gel is a solid mass
- Coarsening – small particles grow initially and act as “nutrients” for bigger crystals

Sol-Gel Process: Drying

- Stage 1 - Constant rate period
 - Decrease in volume of gel is equal to the liquid lost
- Stage 2 - Critical point
 - Network strength is increased due to greater packing
- Stage 3 – Falling rate period
 - Pores have substantially emptied

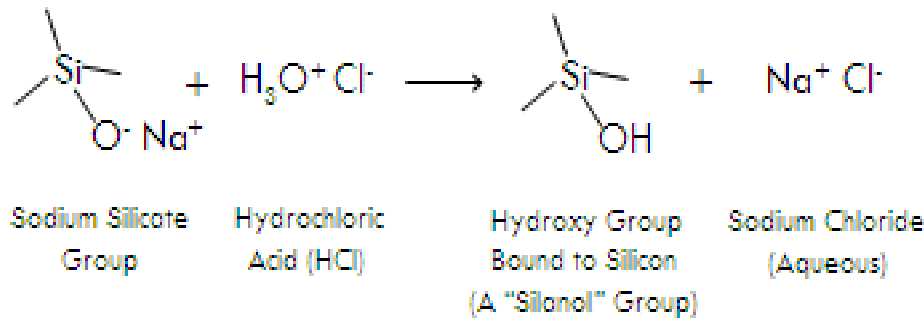
Advantages and Disadvantages Silica Oxides

- Advantages:
 - Straightforward chemistry
 - Easy reactions (water)
- Disadvantages:
 - Somewhat hazardous
 - Expensive

Sodium Silicate

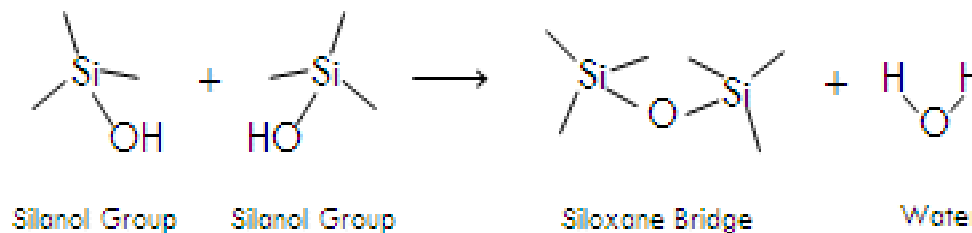
The Two Primary Reactions in Silica Gel Formation via the Waterglass Technique

Hydrolysis



This reaction produces the reactive groups which will form polymer links

Water Condensation



Once silanol groups form, they can condense to form connective silicon-oxygen-silicon bridges

Advantages and Disadvantages Sodium Silicate

- Advantages:
 - Less expensive
- Disadvantages:
 - Sodium silicate molecules do not hydrolyze and condense together when placed in water
 - Resulting gels are fragile and require purification

What Now?

- Sodium Silicate is readily available
- Nissan Chemicals
 - Snowtex
 - Suncolloid
- Aluminum oxide
- Epoxy
- Organically Modified Silicate Films

