

Lecture 7

Sol gel method

In the sol gel process, initially a stable colloidal solution called sol is formed. The sol is a liquid suspension of solid particles ranging in size from 1 nm to 1 micron. It can be obtained by hydrolysis and partial condensation of precursors such as an inorganic salt or a metal alkoxide. The further condensation of sol particles into a three dimensional network produces a gel material. The gel is a diphasic material in which the solids encapsulate the solvent. The molecular weight of the oxide species produced continuously increases. The materials are referred to as aqua sol or aqua gels when water is used as a solvent and aquosol or alcogel when alcohol is used. The general scheme of preparation by sol gel method is shown in Fig. 1

The encapsulated liquid can be removed from a gel by either evaporative drying or with supercritical drying /extraction. The resulting solid products are known as xerogel and aerogel, respectively. When gels are dried by evaporation, the dried product is called xerogel. When the gels are dried by supercritical drying, the dried gel is called aerogels. The aerogel retains high porosity and has very high pore volume.

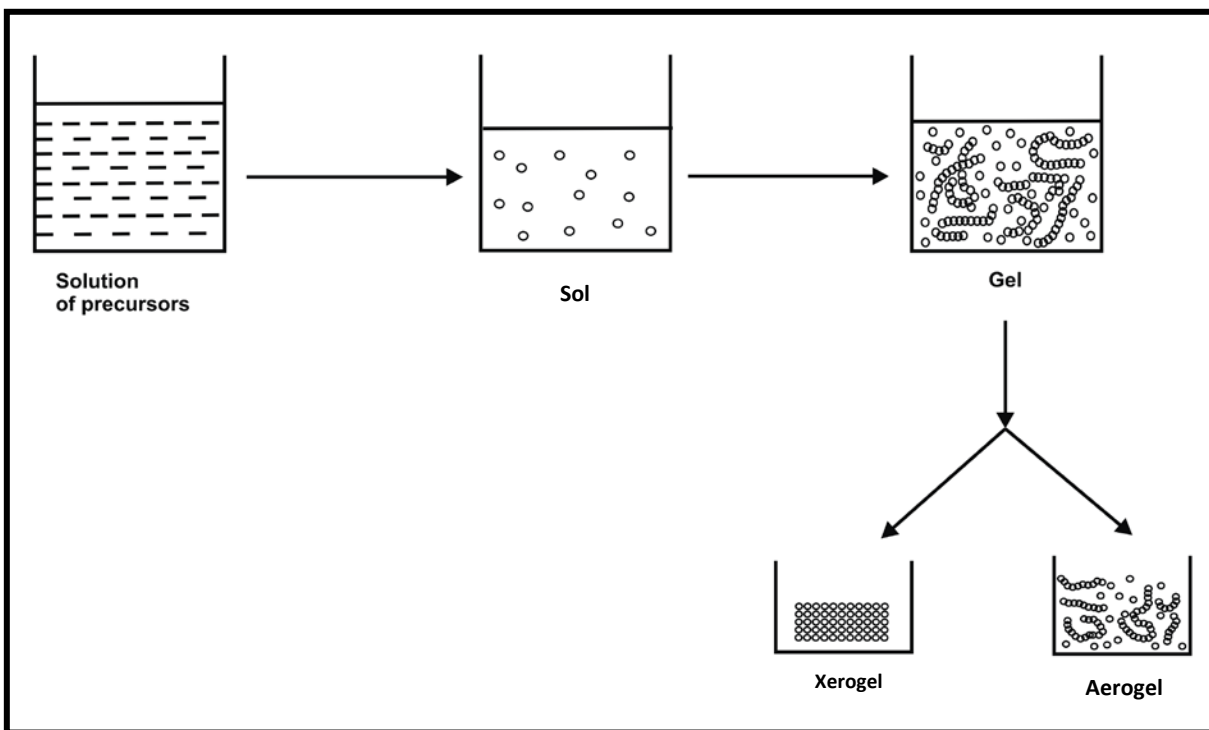


Fig. 1. General scheme of preparation by sol gel method

The sol gel method is distinguished from other routes of material preparation from solutions or melts such as precipitation and crystallization by two main characteristics:

1. Formation of clear colloidal solution due to primary condensation of dissolved molecular precursors.
2. These colloidal particles merge during subsequent gelation stage into polymeric chains by chemical bonding between local reactive groups at their surface.

Both stages are controlled by condensation chemistry that can include as a first step, hydrolysis of hydrated metal ions or metal alkoxides molecules. The condensation chemistry in this case is based on olation/oxolation reactions between hydroxylated species. Olation is a condensation process in which a hydroxyl bridge “–OH–” bridge is formed between two metal centers.

The oxolation is a condensation reaction in which an oxo bridge “-O-” is formed between two metal centers. The steps in sol gel processing are shown in Fig. 2 and discussed below.

Activation and polycondensation : Metal alkoxides are used as precursors in sol-gel operation. Metal alkoxides are most extensively used as these are commercially available in high purity and their solution chemistry is well documented. For preparation of alumina and zirconia, aluminium propoxide and zirconium propoxide are used respectively as precursors.

The metal alkoxides are hydrolysed in alcohol solution containing a controlled amount of water. The sol gel chemistry can be represented by following two reactions :



Where M= metal ; X = H or R (alkyl group)

This is a very simplified representation without giving details of the intermediate or end products. However, this gives an idea of the formation of three dimensional gel network coming from the condensation of partially hydrolyzed species.

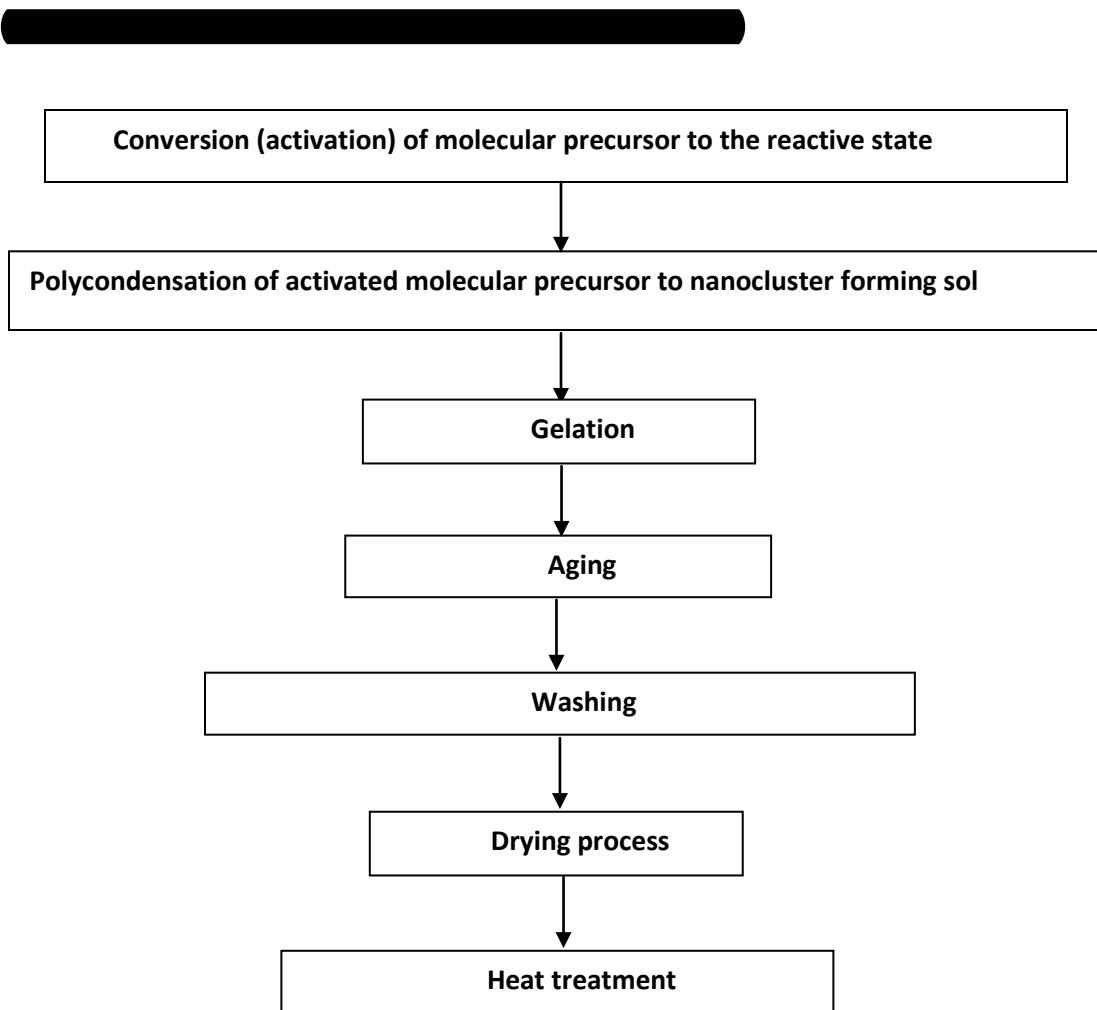


Fig. 2. Basic steps in sol gel processing

Parameters affecting any of the two reactions will affect the properties of the final product. Two of the main parameters that affect are (1) amount and rate of water addition and (2) pH of the solution. The amount of water added is expressed in terms of hydrolysis ratio 'h' and defined as

$$h = \frac{\text{moles of water}}{\text{moles of metal alkoxide } M(OR)_m}$$

When $h < 1$, there is less possibility of forming infinite network because of the presence of few M-OH groups for cross linking and gelation. If excess amount of water is present, that is $h > m$, extensively cross linked gel can be formed. For a given amount of water, another way to control the process is to control the rate of addition of water.