Sol-Gel Process

Spring Semester, 2016

Jinsoo Kim

Department of Chemical Engineering Kyung Hee University

Contents

- I. Introduction
- II. Hydrolysis and Condensation of Non-silicates
- III. Hydrolysis and Condensation of Silicates
- IV. Particulate Sols and their Stability
- V Gelation Phenomena
- VI. Aging of gel
- VII. Drying of gel
- VIII. Structure of Porous Gel
- Sintering IX.
- **Applications of Sol-Gel Process** Χ.

Reference C.J. Brinker, G.W. Scherer, "Sol-Gel Science", Academic Press, Inc., San Diego (1990).

I. Introduction

Colloid Systems:

- one substance is divided into minute particles (colloid particles) and dispersed throughout a second substance
- colloid particles are in the range of 10⁻⁷ m to 10⁻⁹ m (0.1μm 1nm) and can be observed only by electronic scope
- Gravitational forces are negligible and interactions are dominated by shortrange force (e.g. van der Waals force and surface charges)

Suspension: dispersed particles are larger than colloid particles ($> 0.1 \mu m$)

Solution: particles of dissolved substance are of molecular size

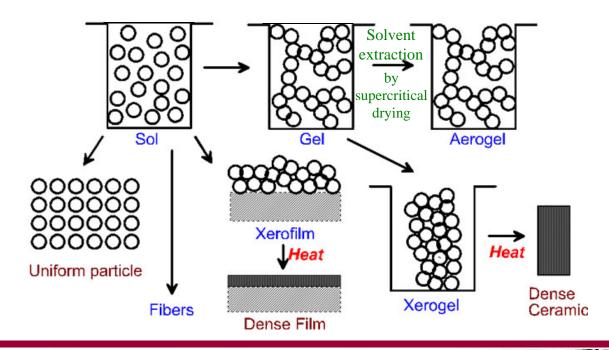
♦ Classification of Colloid systems

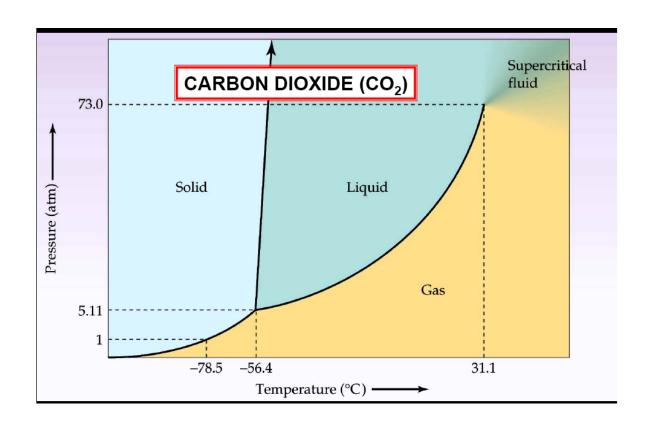
Dispersed	GAS	LIQUID	SOLID
substance			
Medium of dispersion			
GAS		Aerosol (fog)	solid aerosol
			dust in air, smoke
LIQUID	Foam	Emulsion	Sol
	(shaving lather)	(ice cream, oil/water)	
SOLID	Solid foam	Gel	Solid sol
	(alumina foam)	(jelly)	(some alloys)

♦ Sol-Gel Process

Sol-gel processing refers to room temperature formation of solid inorganic materials from molecular precursors in liquid solution. Inorganic salt or metal organic compounds are dissolved in aqueous or organic solvents to form polymeric or particulate sols consisting of fine inorganic particles dispersed in the solvent. These sols are then condensed to various types of gels.

Overview of the sol-gel process





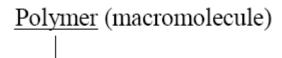
Supercritical drying:

a process of removing the liquid from the pores of wet gel above the critical temperature and critical pressure

♦ Basic Concepts

- Precursors: starting compounds in sol-gel process
- inorganic salts (e.g. AlCl₃)
- metal organic (-M-O-C-) (alkoxide e.g. Al(OC₄H₉)₃) (c.f.) organometallic (-M-C-)
- Ceramic: all metal oxides, carbides, nitrides (both crystalline & non crystalline),
 and other inorganic, nonmetallic materials being through a high temperature process
- Particulate sols: dispersed solid phase consisting of dense (nonporous, nonpolymeric) fine particles with a size larger than 1 nm.

 Polymeric (inorganic) sols: dispersed solid phase contains no dense particles (oxide) larger than 1 nm.



consists of

Hundreds or thousands of

oligomer: molecule of intermediate size, much larger than "mono", but much less than "macro"

Monomer

(capable of forming at least two bonds)

f: functionality of a monomer

 $f=1 \times$

f=2 bifunctional

f = 3 trifunctional

f =4 tetrafunctional

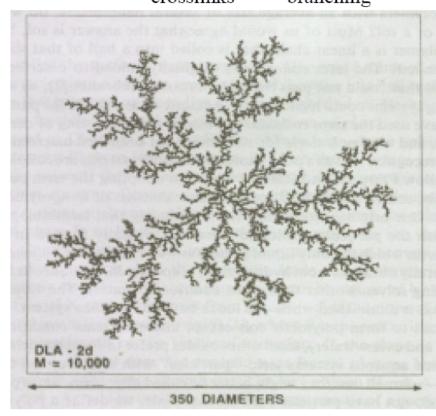
• for bifunctional monomer f = 2,

DIMER: HO-M-O-M-OH

CHAIN: $HO-(M-O)_{n-1}-M-OH$

 $RING \quad : \qquad \quad O\text{-}(M\text{-}O)_{n\text{-}1}\text{-}M$

• for polyfunctional with f>2 crosslinks branching



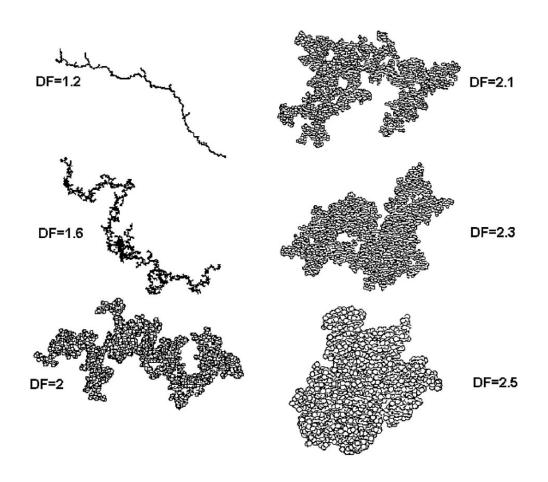
- \Rightarrow fractal polymer
- ** fractal structures:
- monomers (f>2) form bonds in random way
- particulate sol aggregates



- * Mass of dense spherical ball: Mass= $(4/3)\pi R^3 \rho$
- * "Euclidean" object: Mass $\propto R^3$
- * Mass Fractal: Mass $\propto R^{df}$,

 $d_f(mass\ fractal\ dimension)$ < 3

Fractal structures as a function of d_f (mass fractal dimension)



M. Bizi/Natural Science 4 (2012) 372