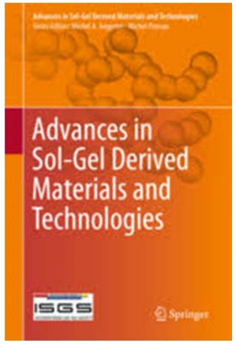
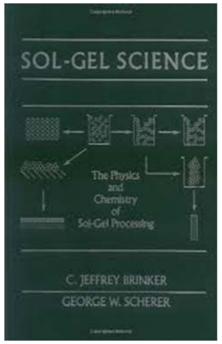
Sol-Gel Methods

Lisa klein

Yi-Jia Chen
National Dong Hwa University





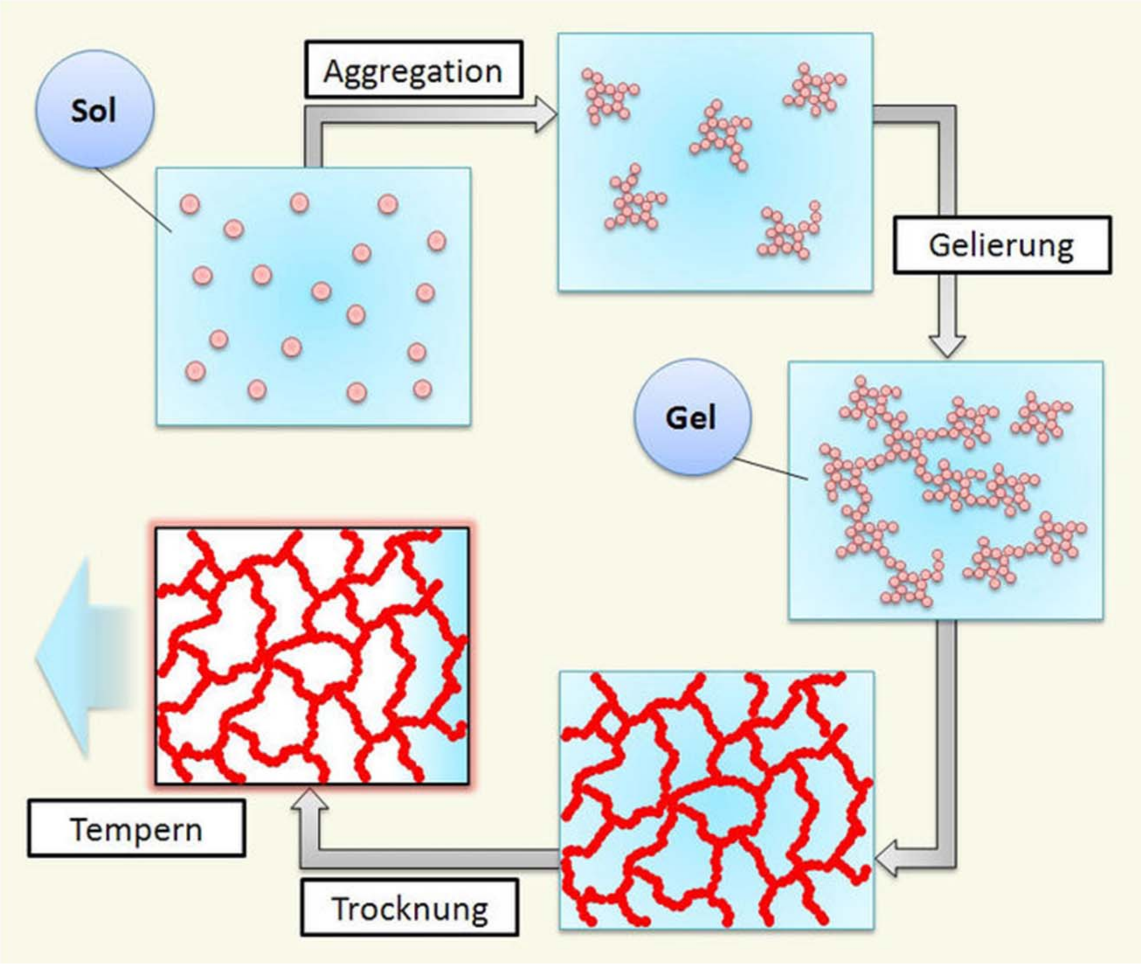
http://www.uio.no/studier/emner/matnat/kjemi/KJM5100/h06/undervisningsmateriale/10KJM5100_2006_sol_gel_d.pdf

Definition of Terms

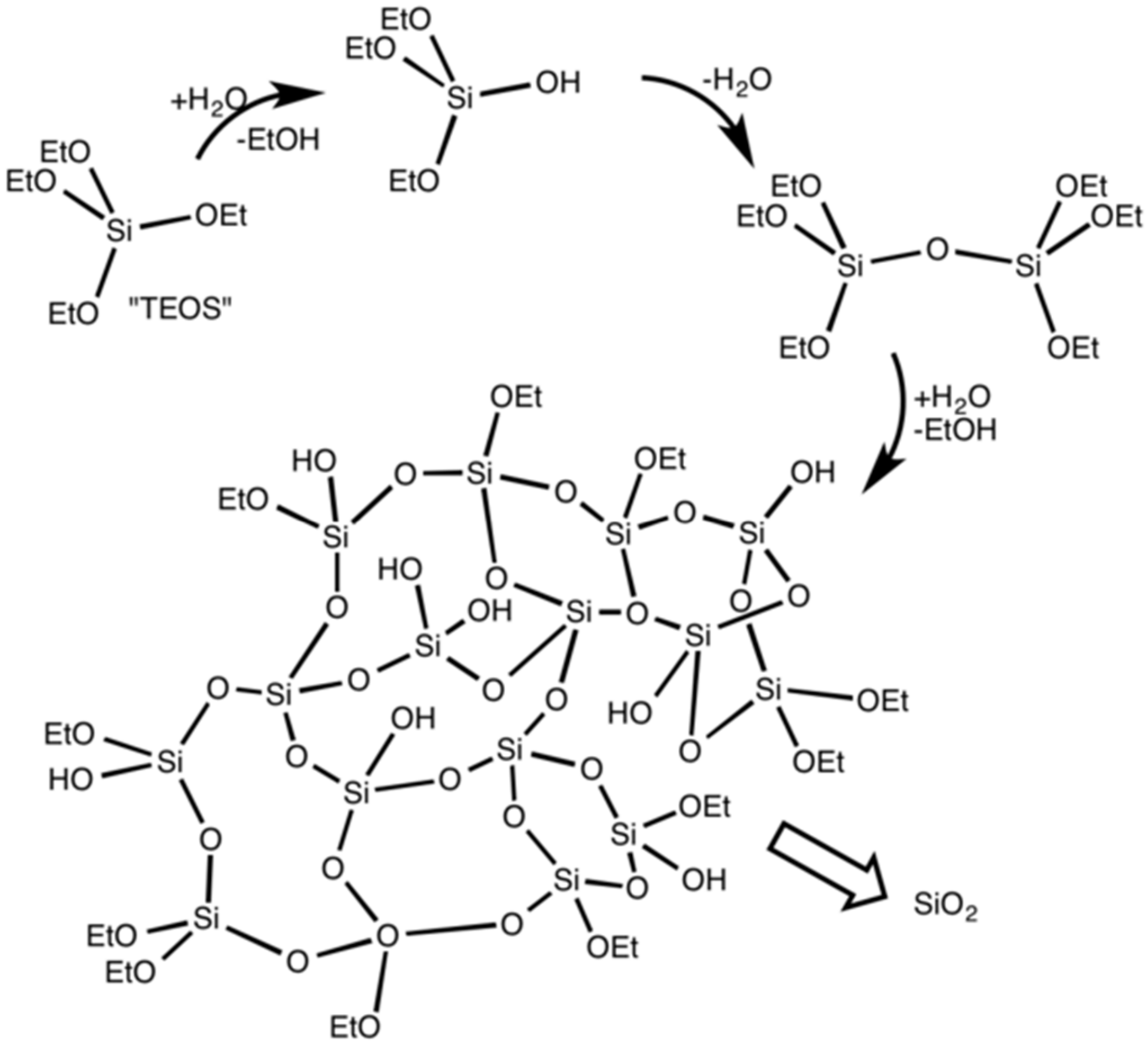
The sol-gel process may be described as:
”Formation of an oxide network through polycondensation reactions of a molecular precursor in a liquid.”

A **sol** is a **stable dispersion of colloidal particles or polymers in a solvent**. The particles may be amorphous or crystalline. An aerosol is particles in a gas phase, while a sol is particles in a liquid,

A **gel** consists of a **three dimensional continuous network**, which encloses a liquid phase, In a colloidal gel, the network is built from agglomeration of colloidal particles.

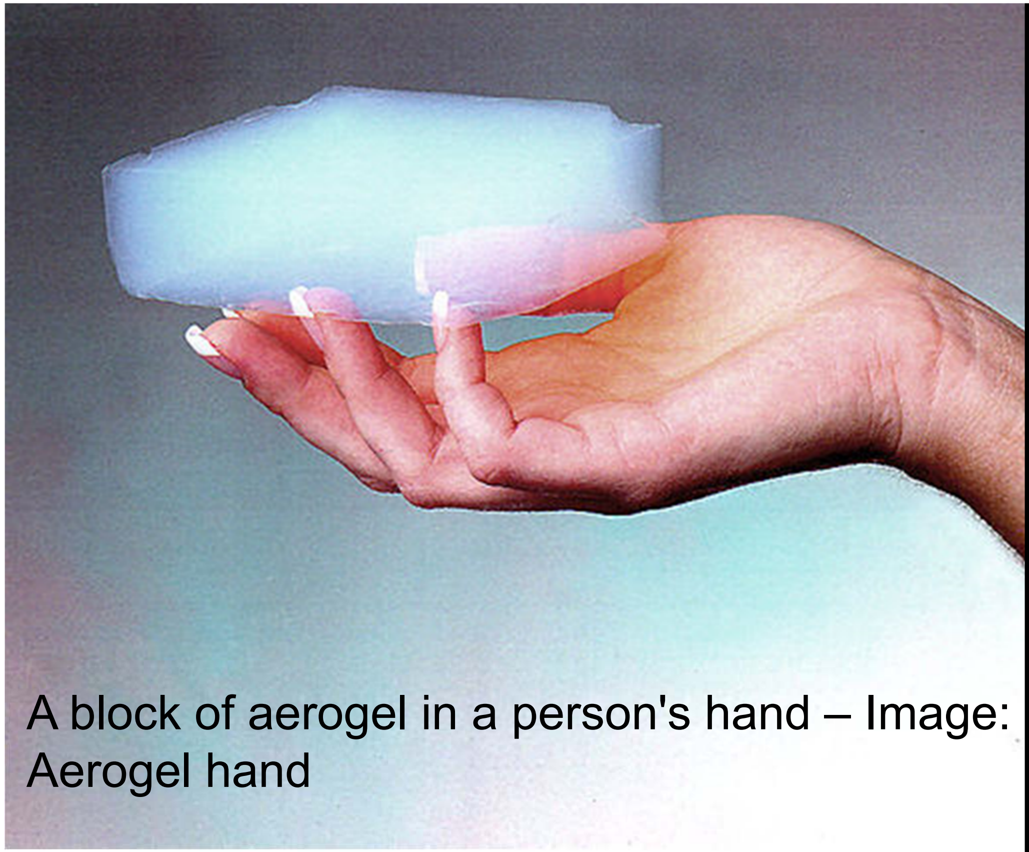


https://wikivisually.com/lang-de/wiki/Sol-Gel-Prozess



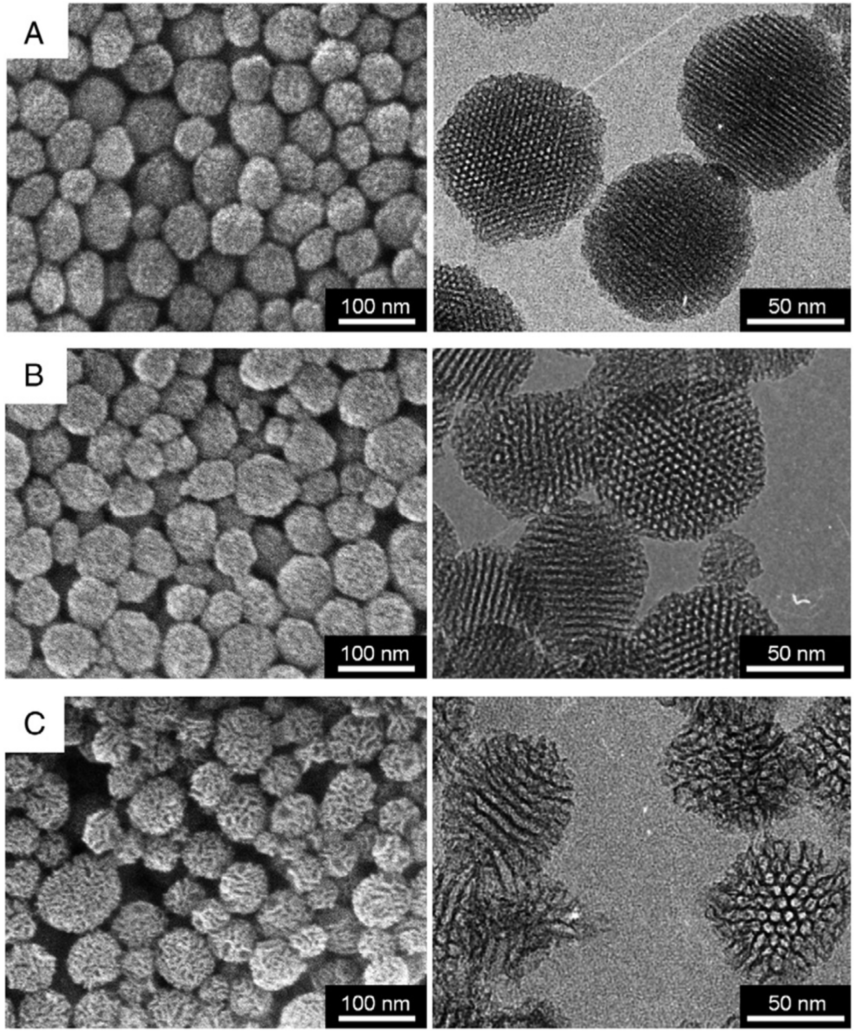
Simplified representation of the condensation of TEOS in sol gel process – Image: Sol Gel Cartoon

https://wikivisually.com/lang-de/wiki/Sol-Gel-Prozess



A block of aerogel in a person's hand – Image: Aerogel hand

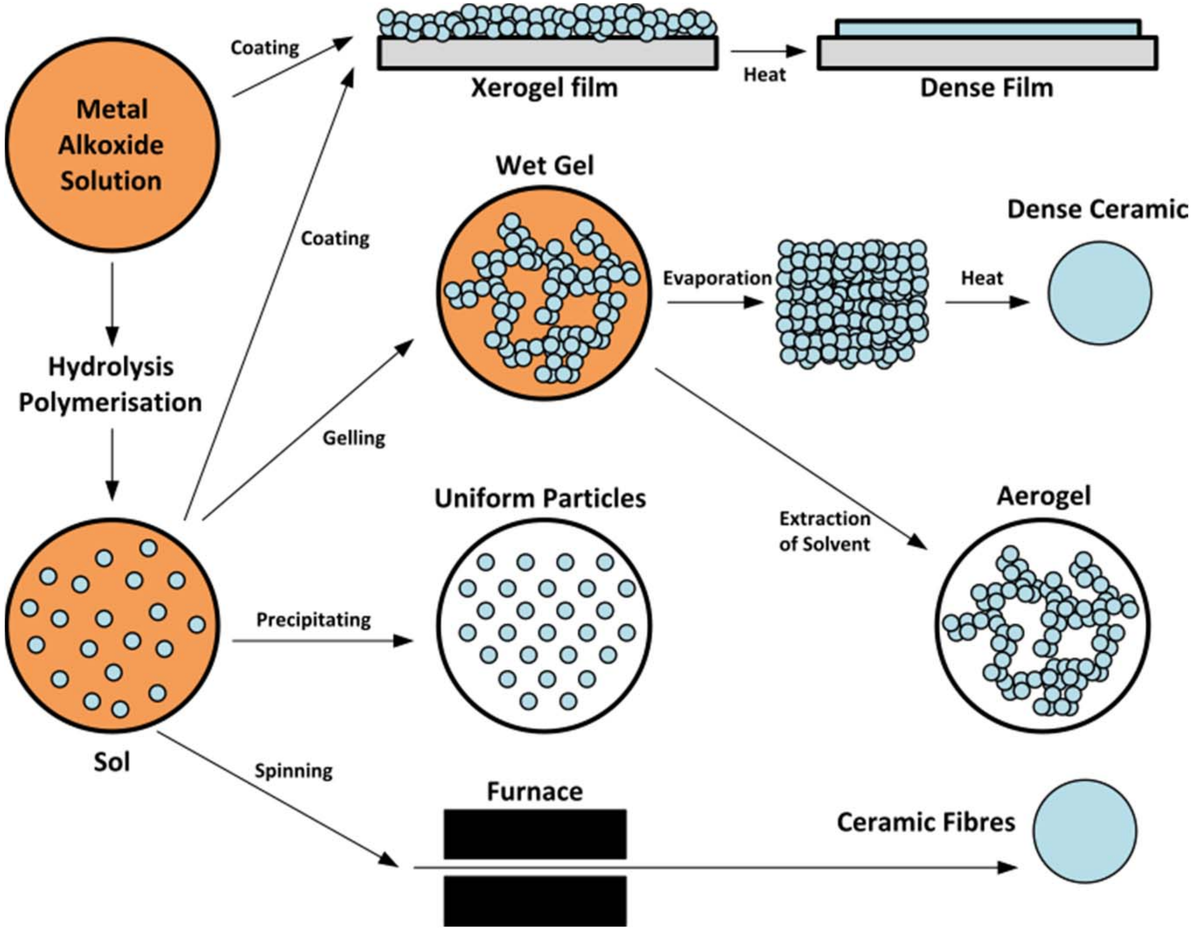
A flower is on a piece of aerogel which is suspended over a flame from a Bunsen burner. Aerogel has excellent insulating properties, and the flower is protected from the flame. – Image: Aerogelflower filtered



FE-SEM (left) and TEM images (right) of TMS-MSNs prepared with TMB/CTAB ratios of (A) 0, (B) 2, and (C) 4.

Hoshikawa Y, Yabe H, Nomura A, Yamaki T, Shimojima A, Okubo T. Mesoporous silica nanoparticles with remarkable stability and dispersibility for antireflective coatings. Chem Mater 2010;22:12–4.

Sol-gel Processing Options



https://commons.wikimedia.org/wiki/File:SolGelTechnologyStages.svg

Motivation of using Sol-Gel Processing

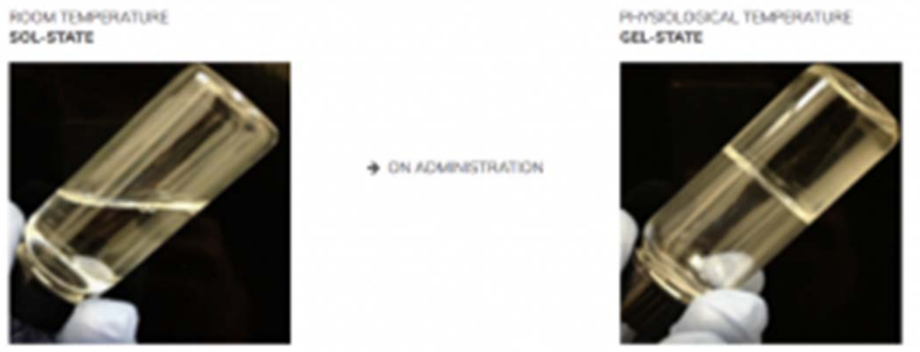
- The idea behind sol-gel synthesis is to “dissolve” the compound in a liquid in order to bring it back as a solid in a controlled manner.
- Multi component compounds may be prepared with a controlled stoichiometry by mixing sols of different compounds.
- The sol-gel method prevents the problems with co-precipitation, which may be inhomogeneous, be a gelation reaction.
- Enables mixing at an atomic level.
- Results in small particles, which are easily sinterable.

The sol-gel method was developed in the 1960s mainly due to the need of new synthesis methods in the nuclear industry. A method was needed where dust was reduced (compared to the ceramic method) and which needed a lower sintering temperature. In addition, it should be possible to do the synthesis by remote control.

Sol-Gel Processing Steps

A sol-gel process occur in 4 steps:

1. Hydrolysis and condensation of molecules. Formation of a sol.
2. Gelation (sol-gel transformation)
3. Ageing
4. Drying



https://investingnews.com/company-profiles/preventative-medicine-biotechnology-cannabidiol-diabetes-obesity-preveceutical-medical/attachment/preveceutical-sol-gel-transition/

Sol

A **sol** consists of a liquid with colloidal particles which are not dissolved, but do not agglomerate or sediment.

Agglomeration of small particles are due to van der Waals forces and a tendency to decrease the total surface energy. Van der Waals forces are weak, and extend only for a few nanometers.

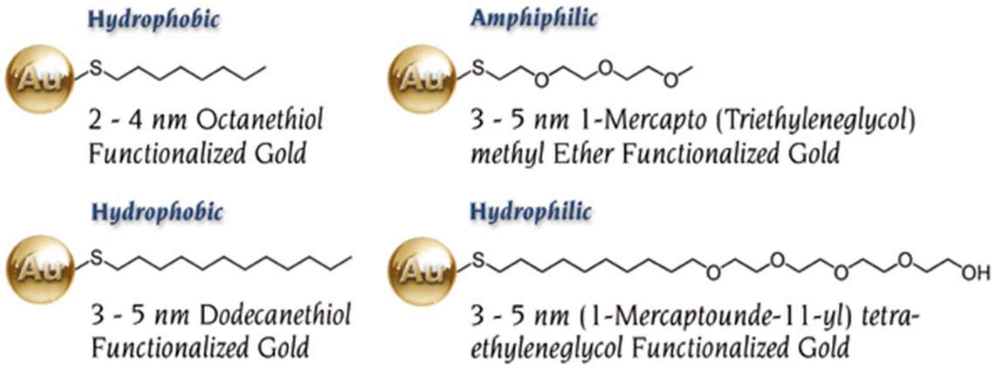
In order to counter the van der Waals interactions, repulsive forces must be established.

May be accomplished by:

Electrostatic repulsion. By adsorption of charged species onto the surface of the particles, repulsion between the particles will increase and agglomeration will be prevented. Most important for colloidal systems.

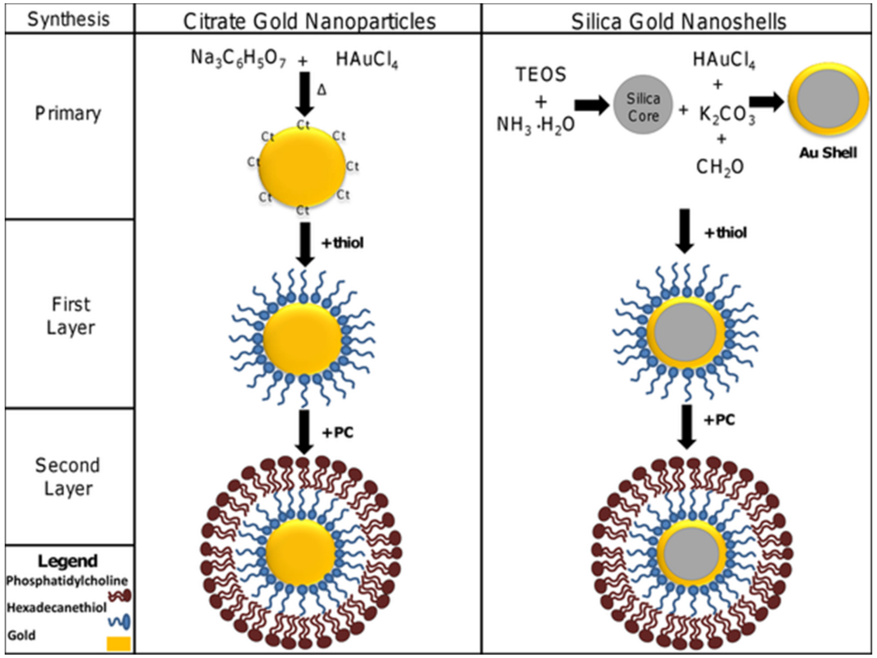
Steric hindrance. By adsorbing a thick layer of organic molecules, the particles are prevented from approaching each other reducing the role of the van der Waals forces. Works best in concentrated dispersions. Branched adsorbates works best. Usual for nanomaterials.

Self-assembled monolayers of thiolates on gold surfaces



http://www.nanoprobes.com/newsletters/Vol10_Iss7.html

Self-assembled monolayers (SAMs)



http://journals.plos.org/plosone/article?id=10.1371/journal.pone.0129172

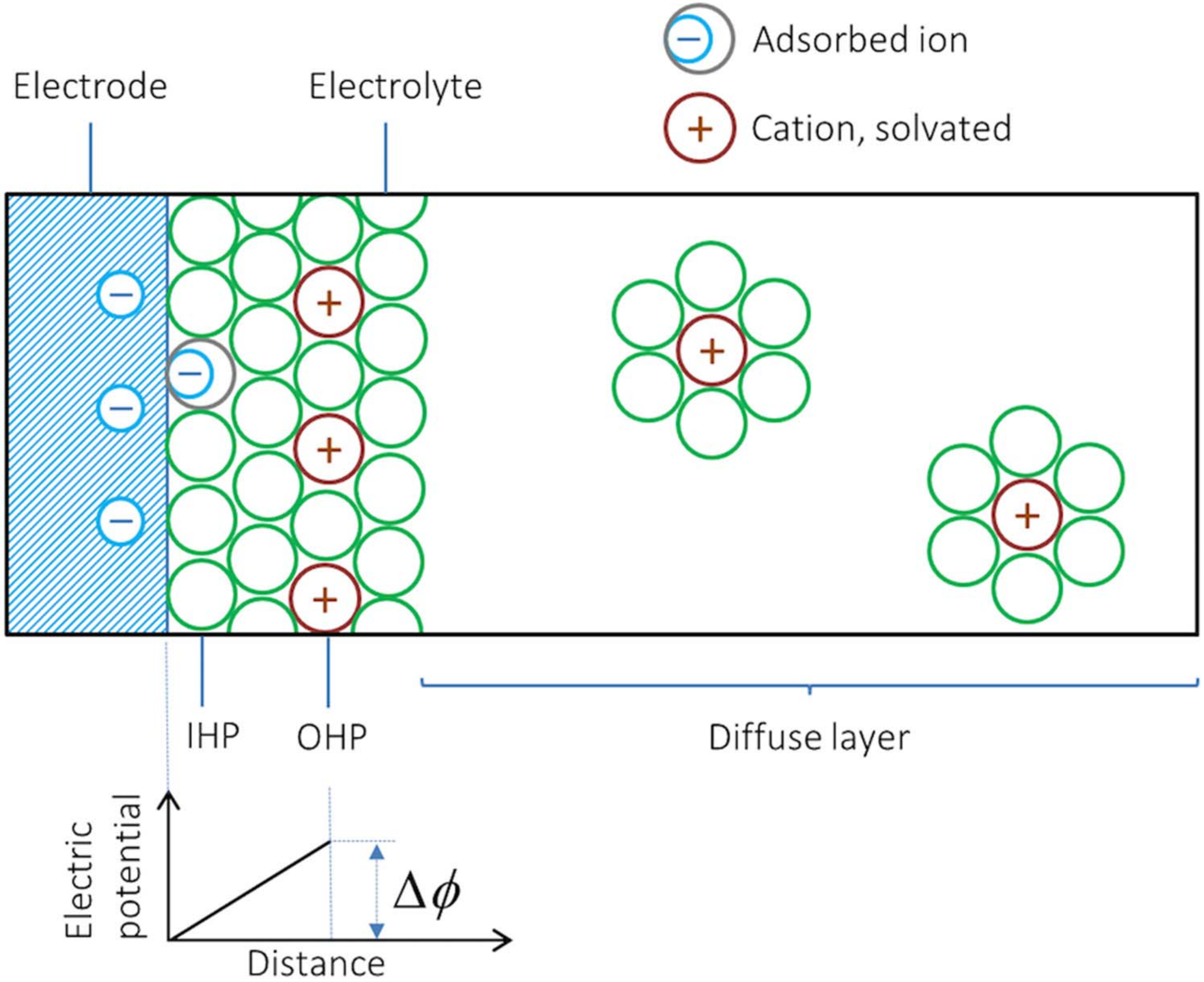
PZC, Point of zero charge

Stabilization due to electrostatic repulsion are due to formation of a double layer at the particle.
The surface of a particle is covered by ionic groups, which determines the surface potential. Counter ions in the solution will cover this layer, shielding the rest of the solution from the surface charges.
For hydroxides the surface potential will be determined by reactions with the ions H^+ and OH^-. Thus, the surface potential is pH dependent.

$$\text{M-OH} + H^+ \rightarrow \text{M-OH}_2^+$$
$$\text{M-OH} + OH^- \rightarrow \text{M-O}^- + H_2O$$

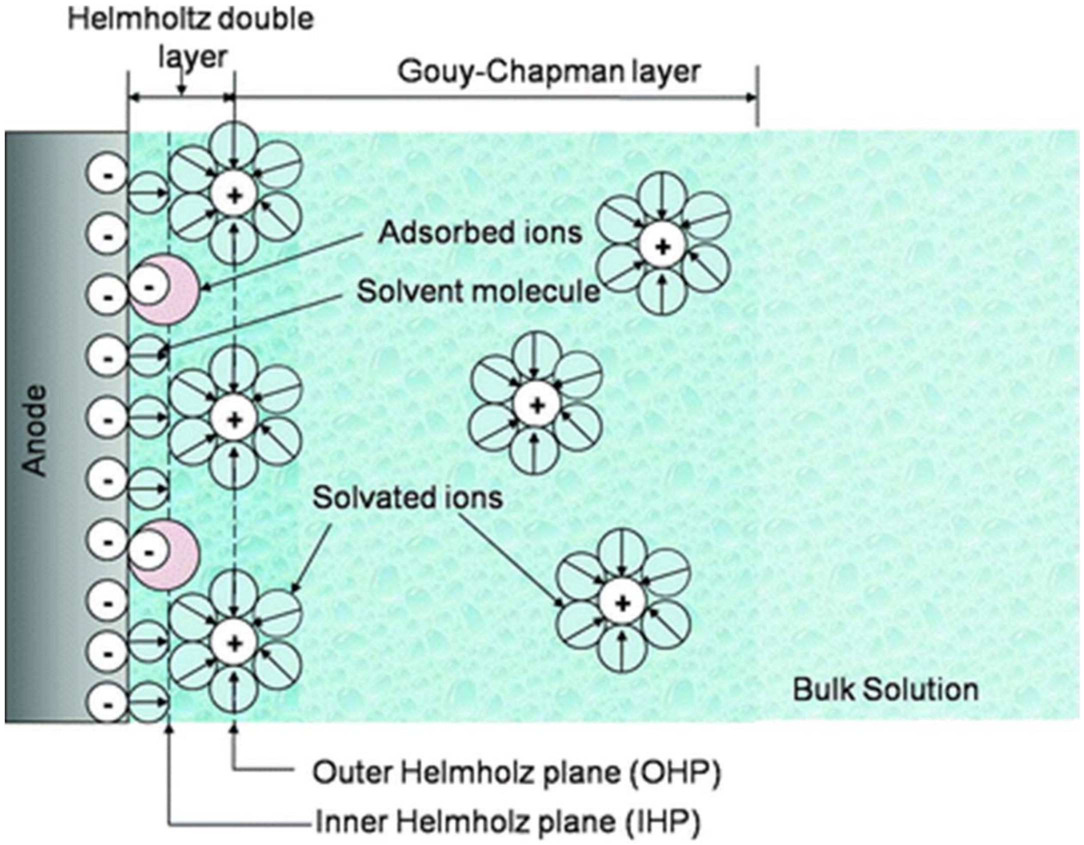
The pH where the particle is neutral is called PZC, point of zero charge.
For **pH > PZC** the surface is **negatively** charged
For **pH < PZC** the surface is **positively** charged.
Typical values: **MgO 12, Al_2O_3 9.0, TiO_2 6.0, SnO_2 4.5, SiO_2 2.5.**
Depends somewhat on the size of the particle and the degree of condensation
The size of the surface potential φ_0 depends on the difference between pH and PZC.

Double Layer in Liquid/Solid Interface



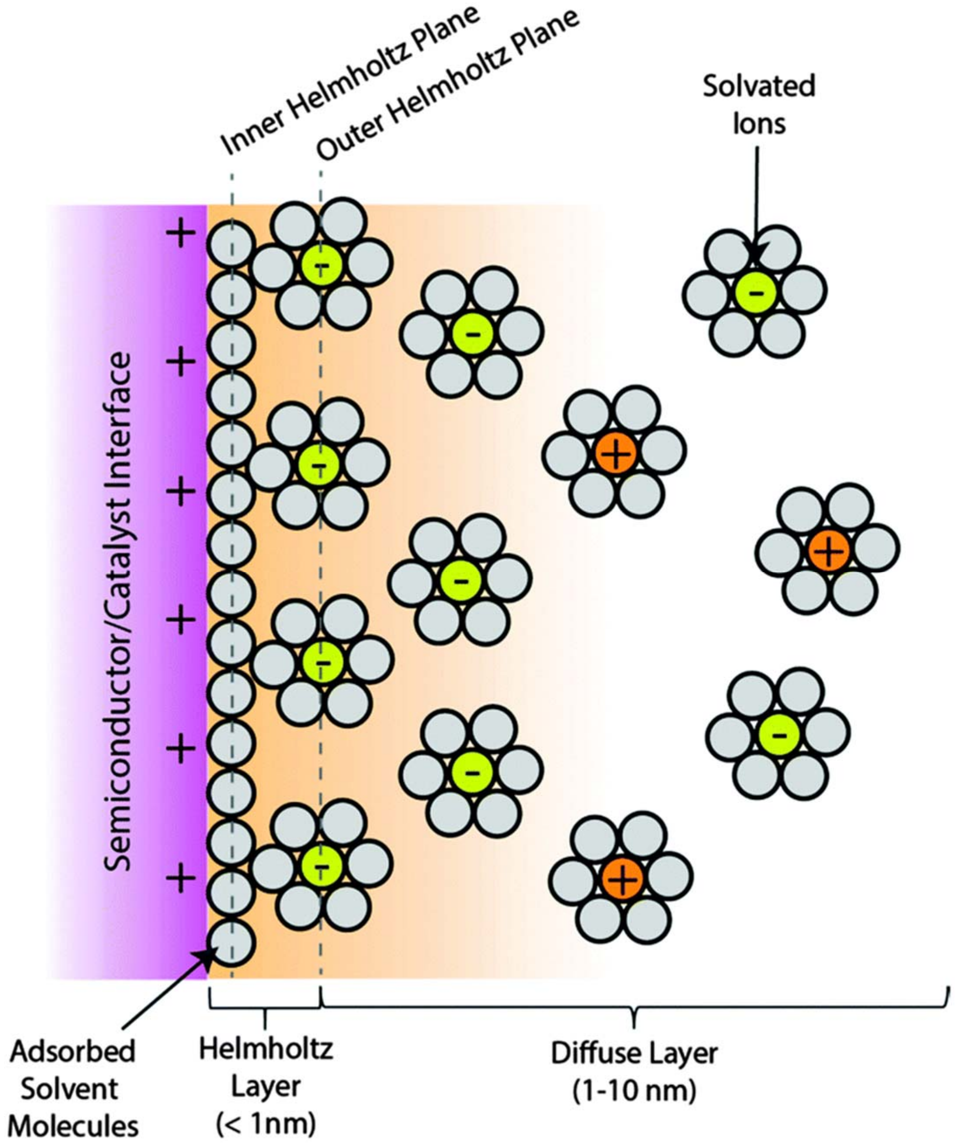
https://www.comsol.com/blogs/does-the-current-flow-backwards-inside-a-battery/

Double Layer in Liquid/Solid Interface



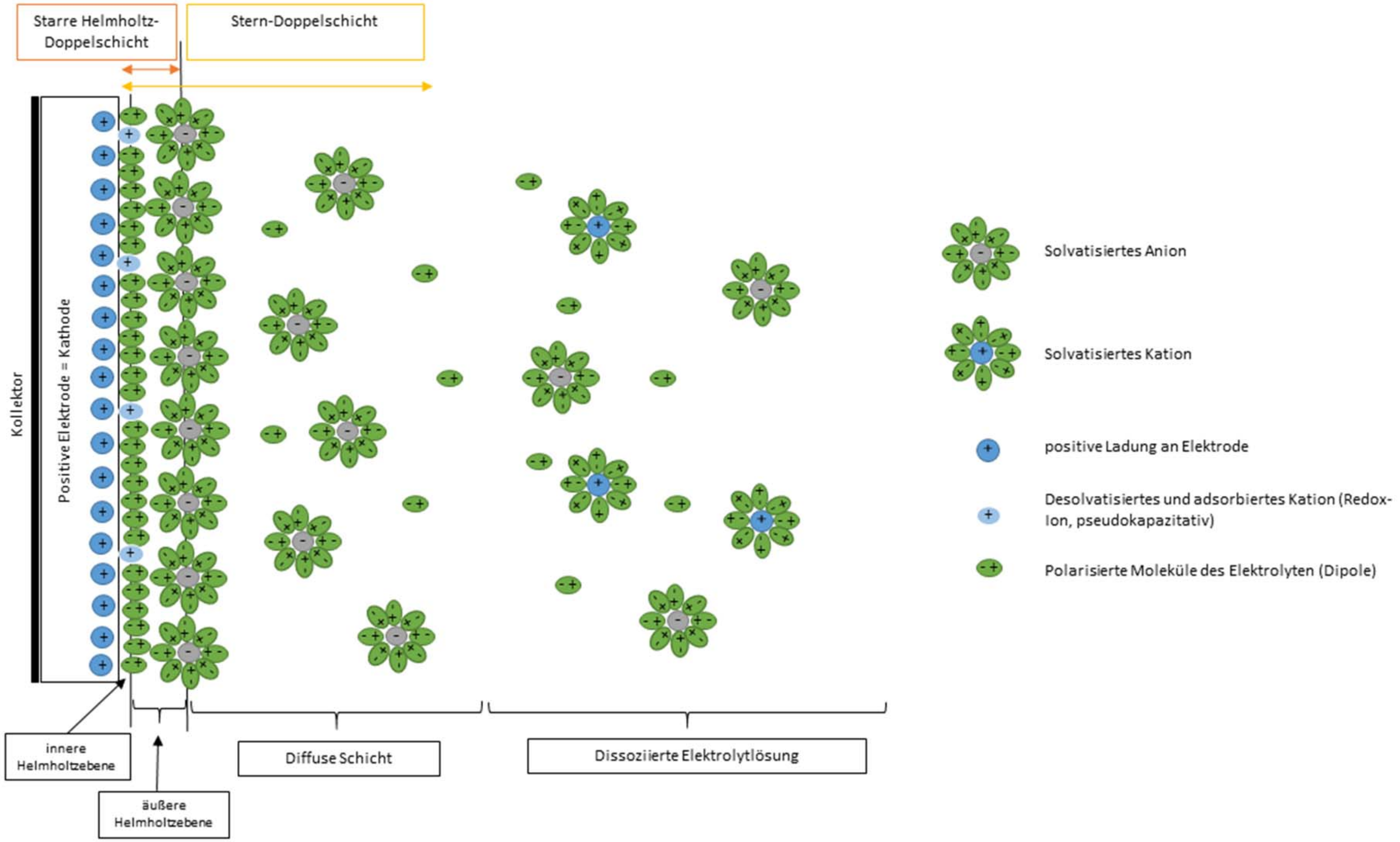
https://link.springer.com/chapter/10.1007/978-3-319-52009-4_8

Double Layer in Liquid/Solid Interface

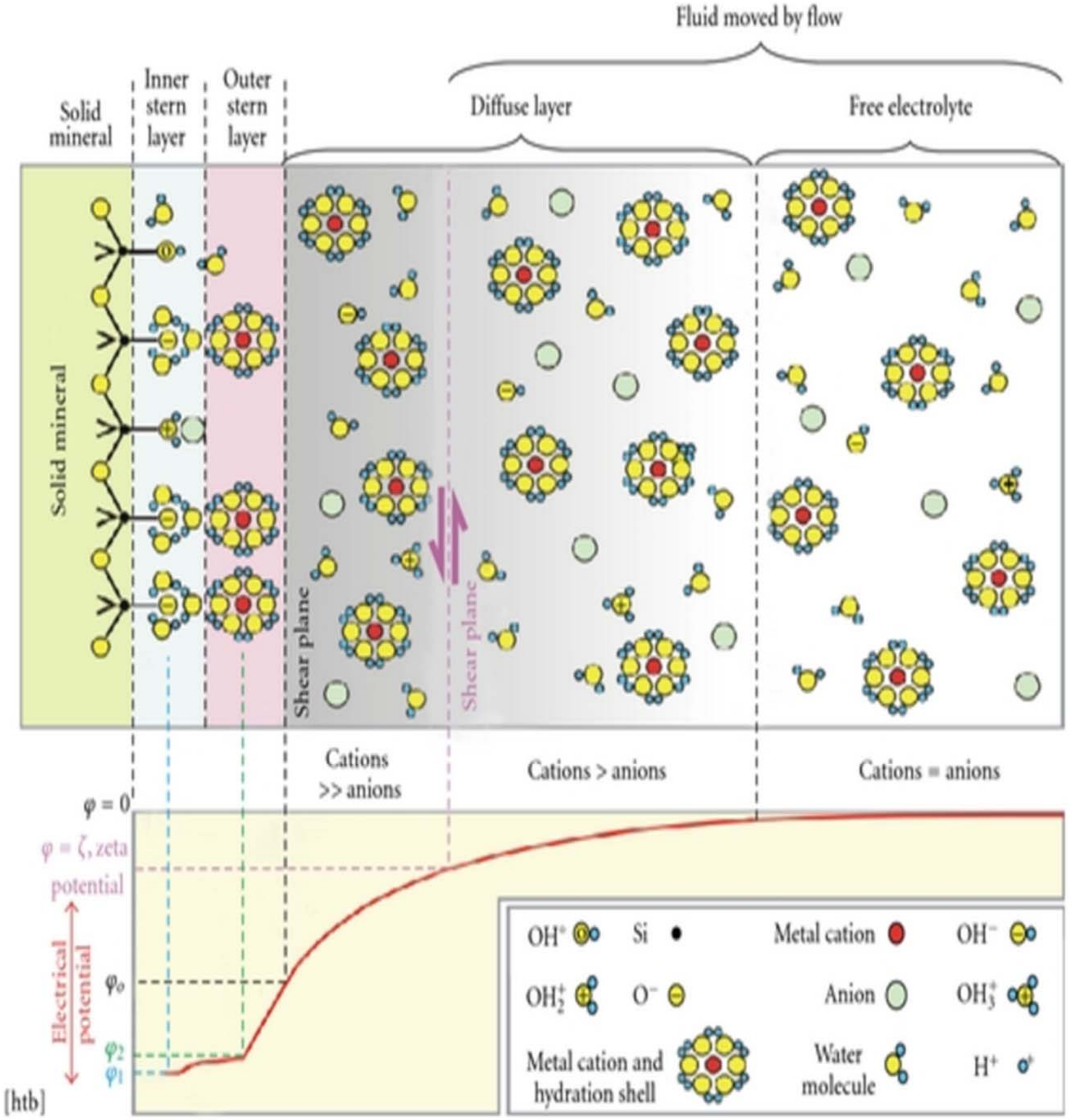


http://pubs.rsc.org/en/content/articlehtml/2016/EE/C5EE03698D

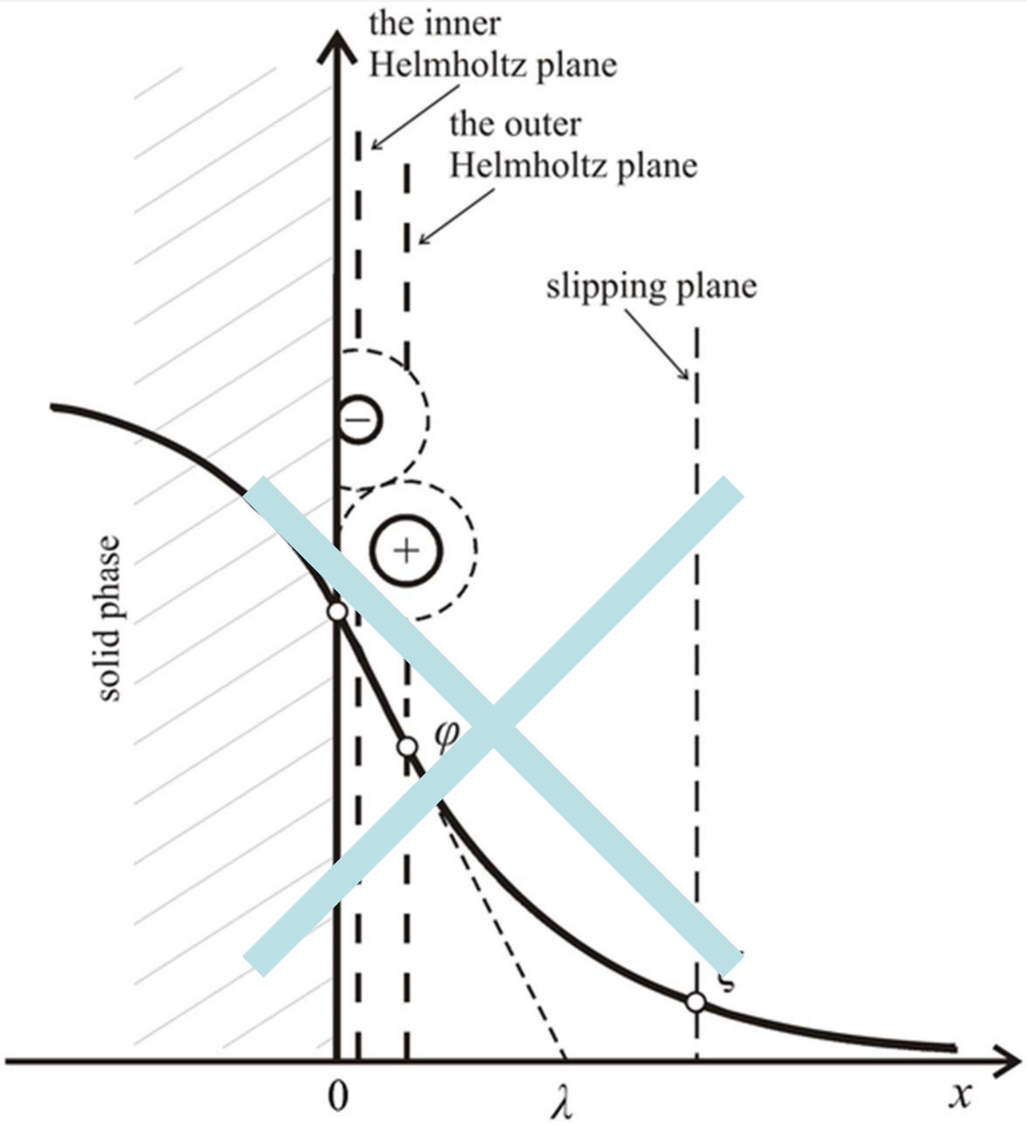
Double Layer in Liquid/Solid Interface



https://www.biooekonomie-bw.de/en/articles/news/biobased-electrode-materials-for-future-energy-storage-systems/



https://www.researchgate.net/figure/Electric-double-layer-first-published-in-81-The-solid-mineral-presented-is-the-case_fig2_257526803



http://elpub.wdcb.ru/journals/rjes/v17/2017ES000591/2017ES000591-fig03.html

Double Layer in Liquid/Solid Interface

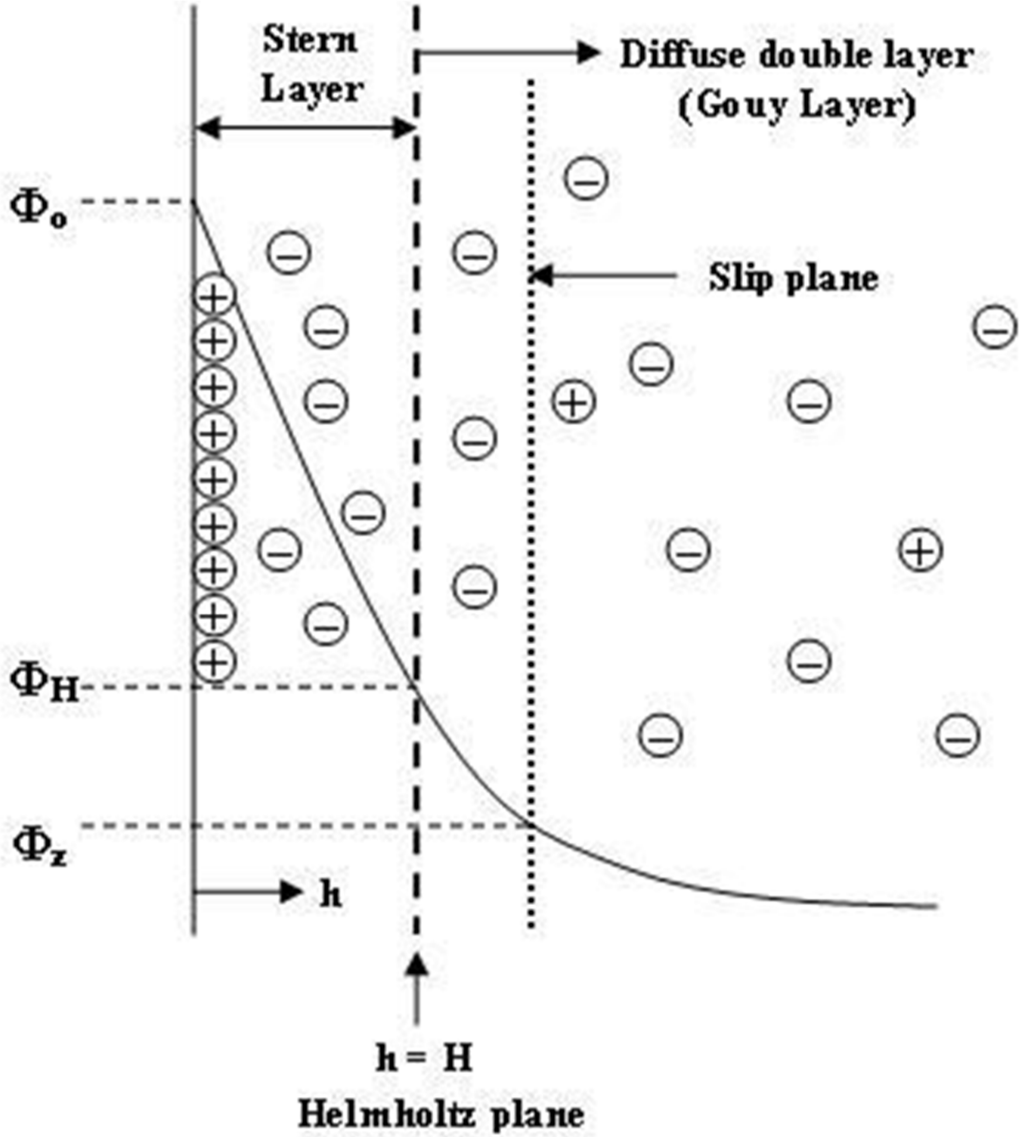
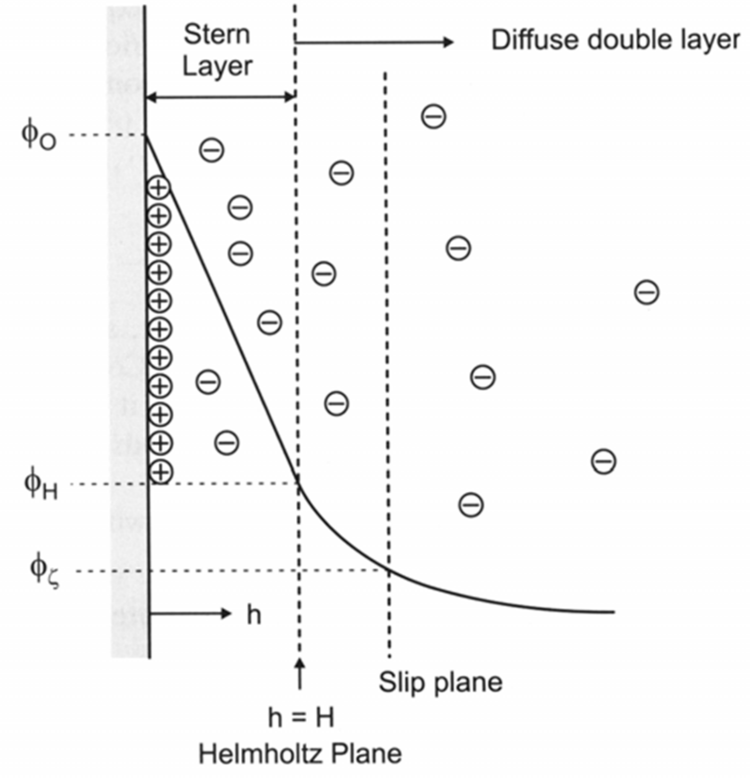


Figure 2.14

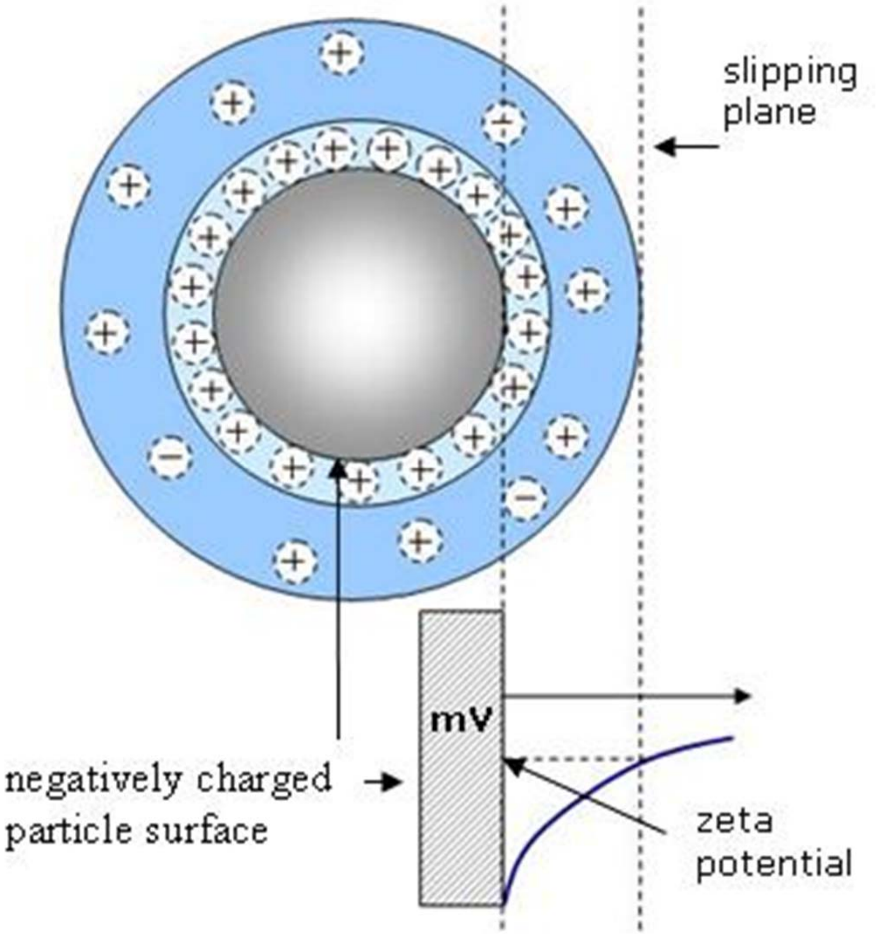
https://depts.washington.edu/solgel/pages/courses/MSE_502/Electrostatic_Stabilization.html

Electrical Field built by Liquid/Solid Interface



- In an electric field the particle will move toward the electrode with the opposite charge. It will carry the adsorbed layer and part of the counter ions.
- The "slip plane" divides the part that moves with the particle and
- the solution. The potential at the slip plane is called the zeta (ζ) potential φ_ζ.
- The pH for which $\varphi_\zeta = 0$ is called the isoelectrical point (IEP)
- The stability of a colloid depends on φ_ζ; the larger the φ_ζ the more stable the colloid. Should be > 30-50 mV.
- Given the same surface potential, the repulsive forces will increase with the particle size.

Zeta potential



The zeta potential is the electric potential in the interfacial double layer (DL) at the location of the slipping plane relative to a point in the bulk fluid away from the interface. In other words, zeta potential is the potential difference between the dispersion medium and the stationary layer of fluid attached to the dispersed particle.

https://www.azom.com/article.aspx?ArticleID=5829

Isoelectrical point (IEP)

The isoelectric point (pI, pH(I), IEP), is the pH at which a particular molecule carries no net electrical charge or is electrically neutral in the statistical mean.

Isoelectric point (IEP) versus point of zero charge (PZC)

- The terms isoelectric point (IEP) and point of zero charge (PZC) are often used interchangeably, although under certain circumstances, it may be productive to make the distinction.
- Electrokinetic phenomena generally measure zeta potential, and a zero zeta potential is interpreted as the point of zero net charge at the shear plane. This is termed the isoelectric point.
- The PZC refers to the absence of any type of surface charge, while the IEP refers to a state of neutral net surface charge.
- The isoelectric point is expected to be somewhat different than the point of zero charge at the particle surface, but this difference is often ignored in practice for so-called pristine surfaces, i.e., surfaces with no specifically adsorbed positive or negative charges.
- In the absence of positive or negative charges, the surface is best described by the point of zero charge. If positive and negative charges are both present in equal amounts, then this is the isoelectric point. (Jolivet J.P., Metal Oxide Chemistry and Synthesis. From Solution to Solid State, John Wiley & Sons Ltd. 2000, ISBN 0-471-97056-5 (English translation of the original French text, De la Solution à l'Oxyde, InterEditions et CNRS Editions, Paris, 1994).

堆　　散

Coagulation/flocculation

Coagulation of a sol may occur if:

- The surface potential (φ_0) is lowered (by changing pH)
- By increasing the number of counter ions. An increase in the concentration of counter ions result in a decrease of the thickness of the double layer.

In some cases a coagulated colloid may be re-dispersed. This is called "**peptizing**" This is done e.g. by removing the surplus counter ions by washing, or by adding charged ions, so that the double layer is restored.

Four (4) Sol-Gel Processing Steps

Nomenclature of Materials

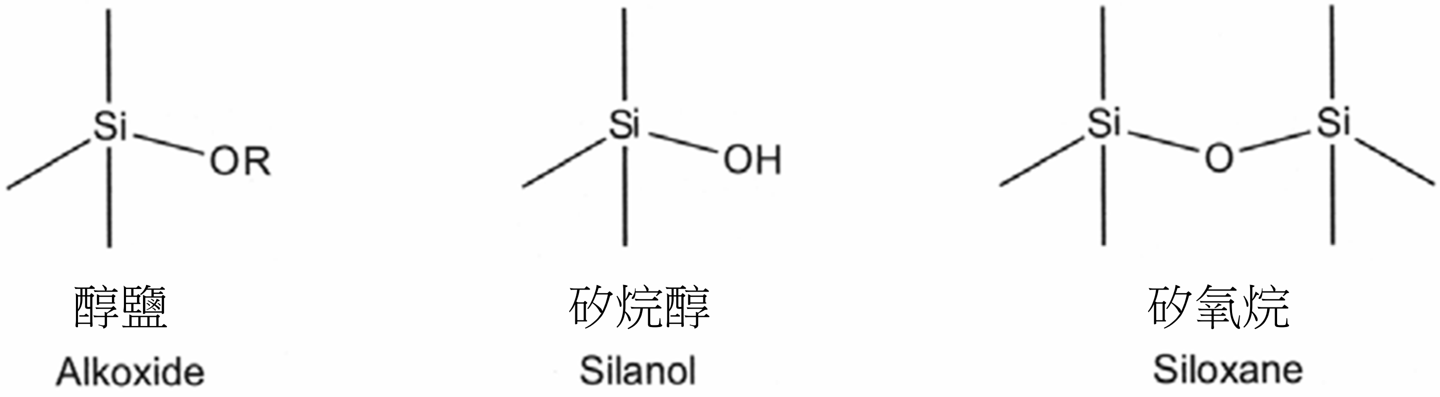


Figure 4-48. Silicon–oxygen groups relevant to sol–gel processing.

The starting point for formation of a silica gel may be alkoxides or silanols. These are reacted to siloxane groups.

Hydrolysis and Condensation

$$\equiv\text{Si–OR} + \text{H}_2\text{O} \longrightarrow \equiv\text{Si–OH} + \text{ROH}$$

Hydrolysis

$$\equiv\text{Si–OH} + \equiv\text{Si–OR} \longrightarrow \equiv\text{Si–O–Si}\equiv + \text{ROH}$$

$$\equiv\text{Si–OH} + \equiv\text{Si–OH} \longrightarrow \equiv\text{Si–O–Si}\equiv + \text{H}_2\text{O}$$

Condensation

The alkoxide is dissolved in an organic solvent, usually alcohol.

Gelation starts with a change in pH (in water based systems) or by addition of water (in alcohol based systems).

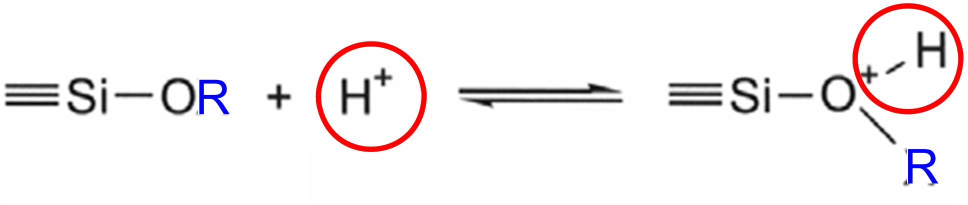
Two reaction paths; acidic and basic environments:

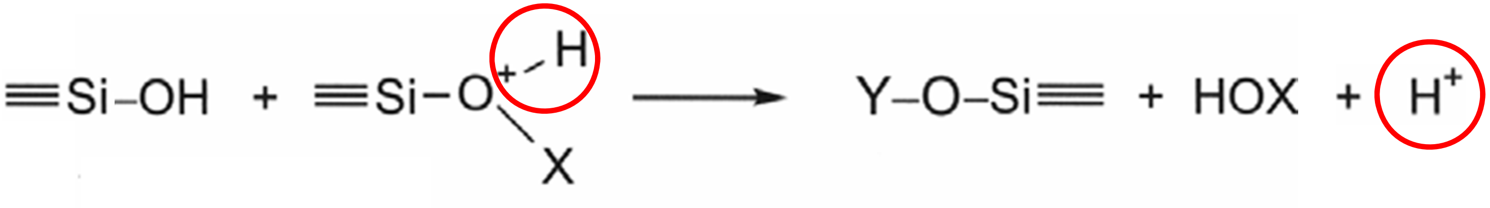
- At pH < PZC the species are positively charged
- At pH > PZC the species are negatively charged

for Silica Gel

Reactions in acidic environments

The oxygen atom in Si-OH or Si-OR is protonated and H-OH or H-OR are good leaving groups. The electron density are shifted from the Si atom, making it more accessible for reaction with water (hydrolysis) or silanol (condensation).



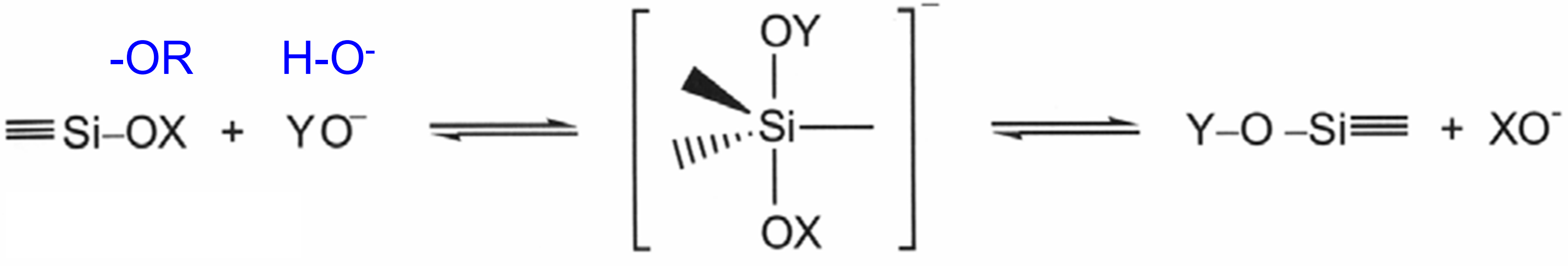


hydrolysis reaction: **X = R, Y = H**

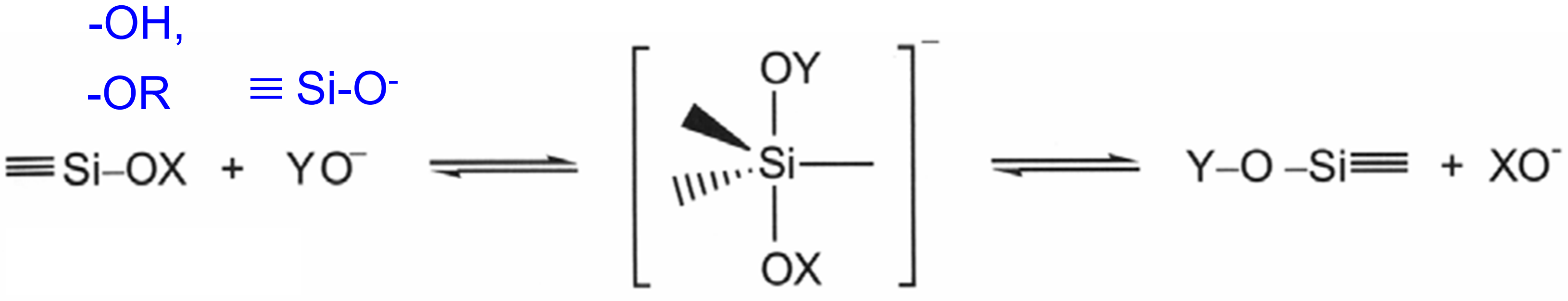
condensation reaction: **X = R or H, Y = Si≡**

Reactions in basic environments

Nucleophilic attack by OH^- or $Si\text{-}O^-$ on the central Si atom. These species are formed by dissociation of water or Si-OH.
The reactions are of S_N2 type where OH- replaces OR- (hydrolysis) or silanolate replaces OH^- or OR^- (condensation).

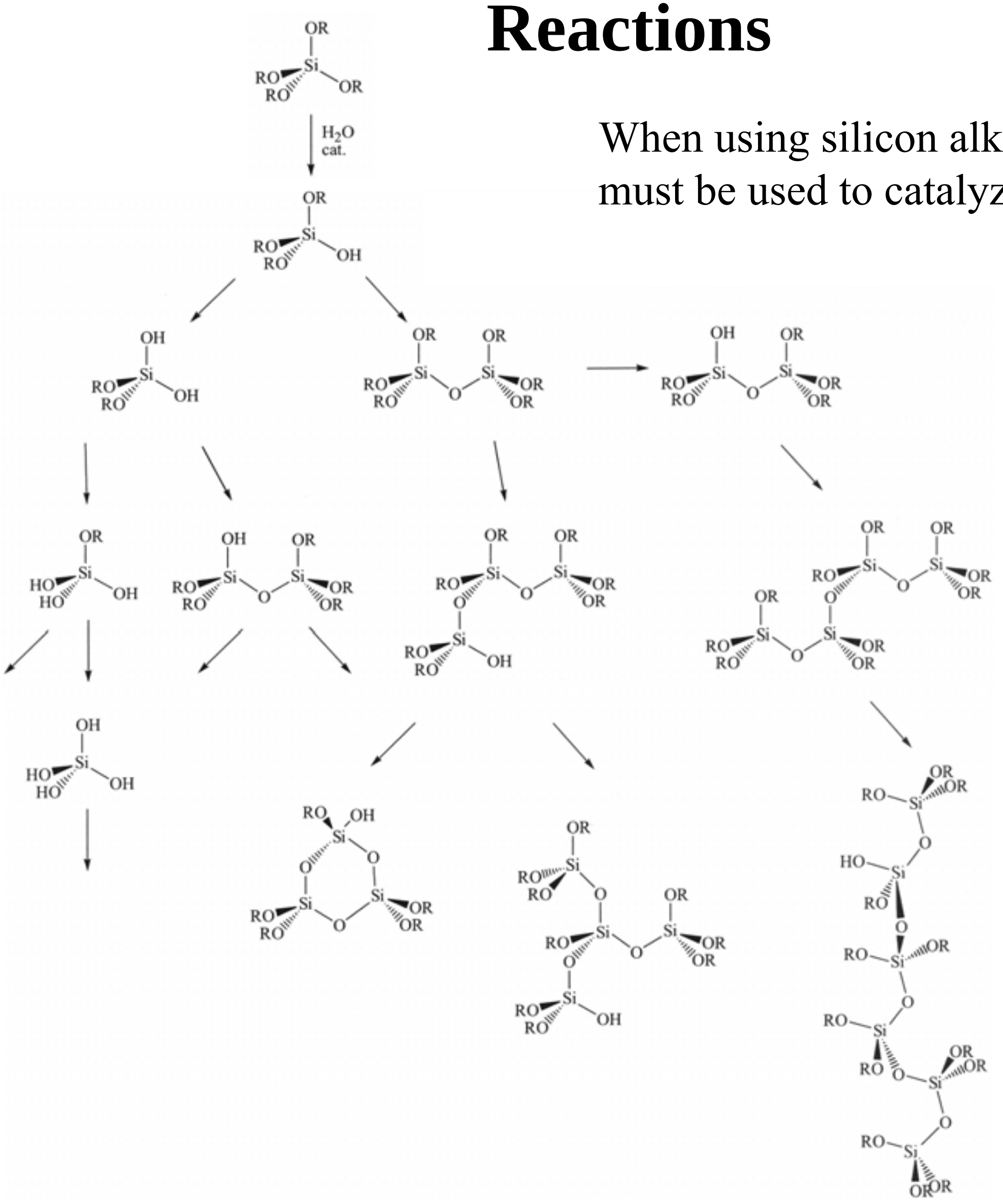


X = R, H   Y = H, ≡Si



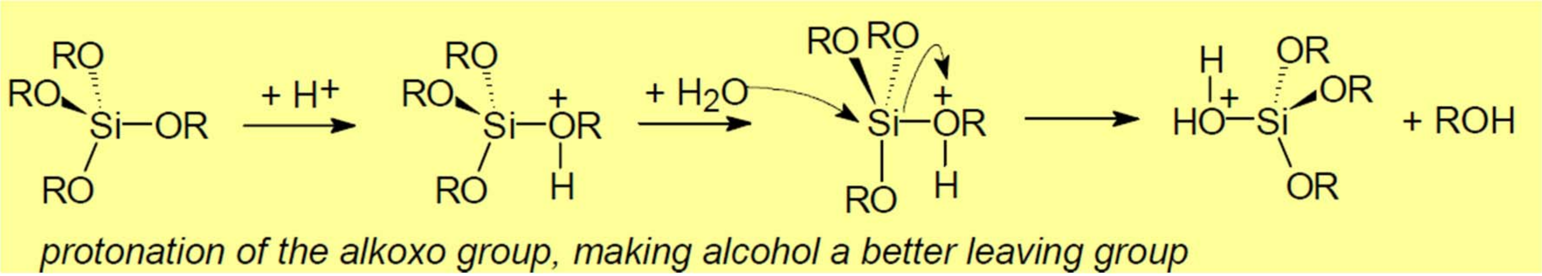
Reactions

When using silicon alkoxides, acid or base must be used to catalyze the reactions.



Acidic Conditions

Under acidic conditions (e.g. with mineral acids), the hydrolysis reaction is speeded up more efficiently than the condensation reaction :

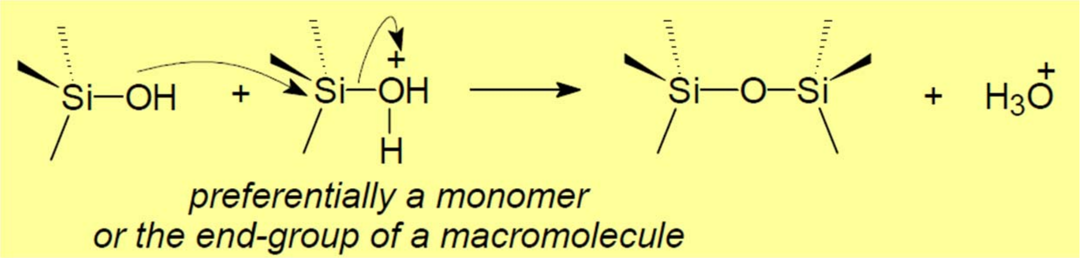


protonation of the alkoxo group, making alcohol a better leaving group

Condensation involves the attack of silicon atoms carrying protonated silanol species by neutral ≡Si-OH nucleophiles. 親核體

- acidic conditions further the formation of protonated silanol species, but inhibit some nucleophiles
- the most basic silanol species (the most likely to be protonated) are those contained in monomers or weakly branched oligomers :

單體 短聚合體



preferentially a monomer
or the end-group of a macromolecule

- So a bushy network of weakly branched polymers is obtained

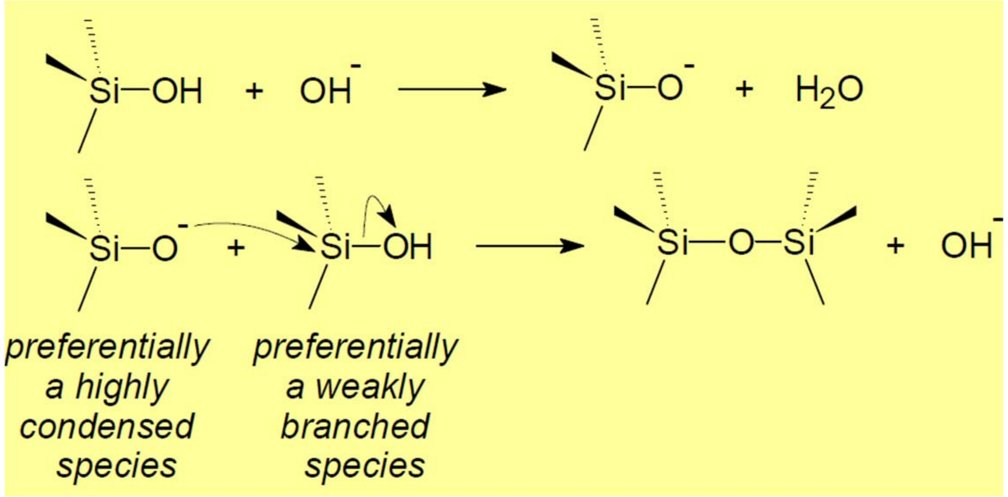
Basic Conditions

Under basic conditions (e.g. with ammonia), hydroxyl anions (OH^-) and deprotonated silanol (≡Si-O^-) are better nucleophiles than water and silanol species.

- a fast attack at the silicon atom and both hydrolysis and condensation reactions occur simultaneously.

The condensation involves the attack of a deprotonated silanol (≡Si-O^-) on a neutral siloxane species.

- the acidity of silanol increases when -OH or -OR groups are replaced with $^-$O-Si≡ groups because of the reduced electron density on Si atom.



- The result of basic catalysis is an **aggregation** (monomercluster) that leads to more compact highly branched silica networks, that are not interpenetrable before drying and thus behave as discrete species.

Condensation

The condensation process is dynamic, and may be steered in the desired direction by adjusting the proper parameters. Parameters which influences the condensation process:

1. Type of precursor (steric hinderance)
2. The ratio between alkoxide and water (R_W)
3. Type of catalyst used
4. Type of solvent
5. Temperature
6. pH
7. Relative and absolute concentrations of the reactants.

1. Type of precursor

- The stability and reactivity of the silicon alkoxides are influenced by a steric factor. Bulky ligands slow down the hydrolysis:

Reactivity: $Si(OMe)_4 > Si(OEt)_4 > Si(O^nPr)_4 > Si(O^iPr)_4$

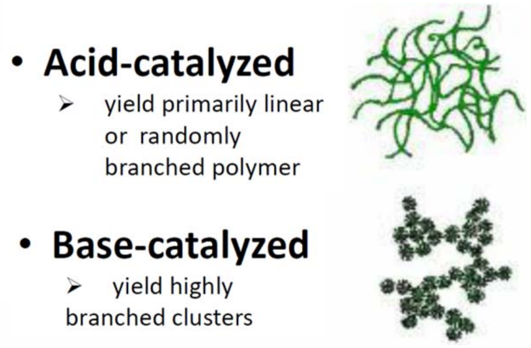
n-propoxide isopropoxide
正丙醇矽 異丙醇矽

- The electron density on Si will influence the reaction rate.

$Si\text{-}R > Si\text{-}OR > Si\text{-}OH > Si\text{-}O\text{-}Si$

Acid catalyzed reaction demands high electron density
Base catalyzed reaction demands low electron density

This results in:
Acid catalyzed: more straight chains
Base catalyzed: more branched network



- **Acid-catalyzed**
  - yield primarily linear or randomly branched polymer
- **Base-catalyzed**
  - yield highly branched clusters

http://www.altazinitiative.org/Word%20Documents/PdfsLBConf.2010/Hawaii.LBA2010-11.IntroSol-GelProcess-Brodhacker2.pdf.pdf

2. The ratio between alkoxide and water (R_W)

The water ratio, R_W. (OR/H_2O)

$$Si(OR)_4 + 2H_2O \rightarrow SiO_2 + 4ROH$$

The reaction states that a water ratio of R_W = 2 (OR/H_2O) is needed to convert everything to SiO_2. A water ratio of R_W=1 leads to complete hydrolysis but no condensation.

Increasing the water content (i.e. lower R_W) will reduce condensation.

Reducing the water content increases the condensation.

3. Type of catalyst used

The nucleophilic activation by Lewis bases (dimethylaminopyridine, n-Bu_4NF, NaF) is also efficient for both hydrolysis and condensation reactions, which occur simultaneously as for basic catalysis.

- reversible formation of a pentavalent intermediate with F^- that stretches and weakens the surrounding ≡Si-OR bonds.
- the positive charge of the silicon atom increases, rendering the silicon atom more prone to nucleophilic attack.
- Highly branched silica networks are obtained.

4. Type of solvent

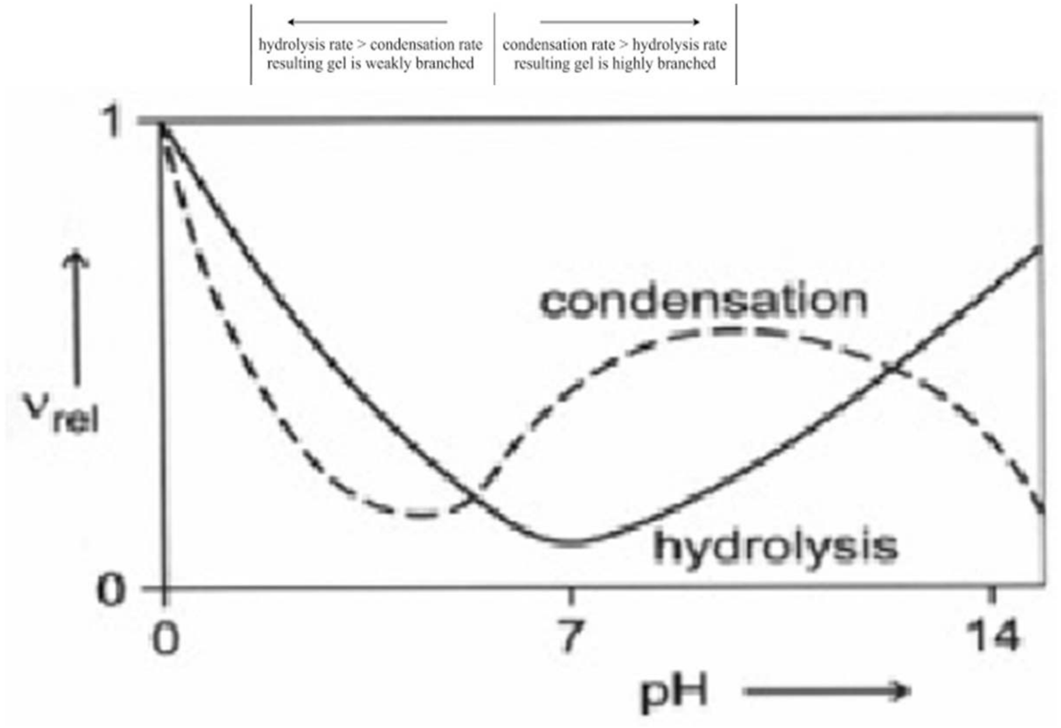
Solvent: The polarity, dipolar moment, viscosity, protolytic/non protolytic properties are important for the reactions taking place.

- Polar solvents stabilize polar gels by hydrogen bonding.
- Non-polar solvents are better for systems which are not completely hydrolyzed.

6. pH

pH: The dependence of the reaction rate with pH is different for acid and base catalyzed systems.

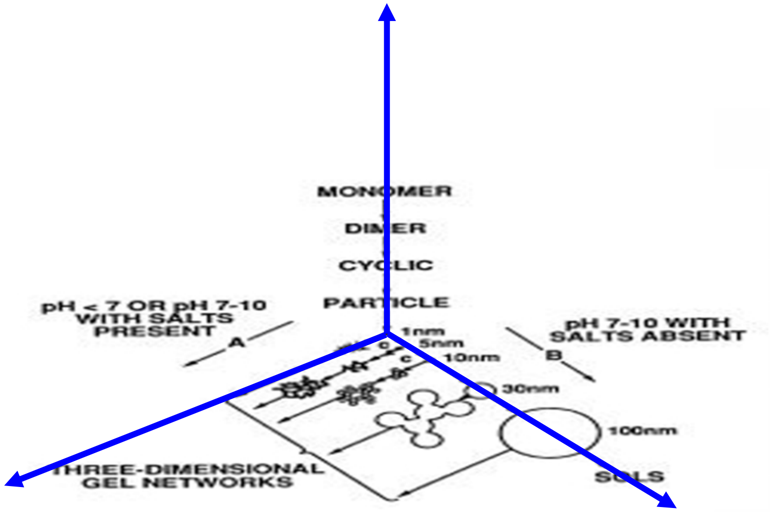
A minimum for hydrolysis is observed at pH = 7 and for condensation at pH = 4.5 (IEP for silica). The sol particles may be reached without an electrostatic barrier.

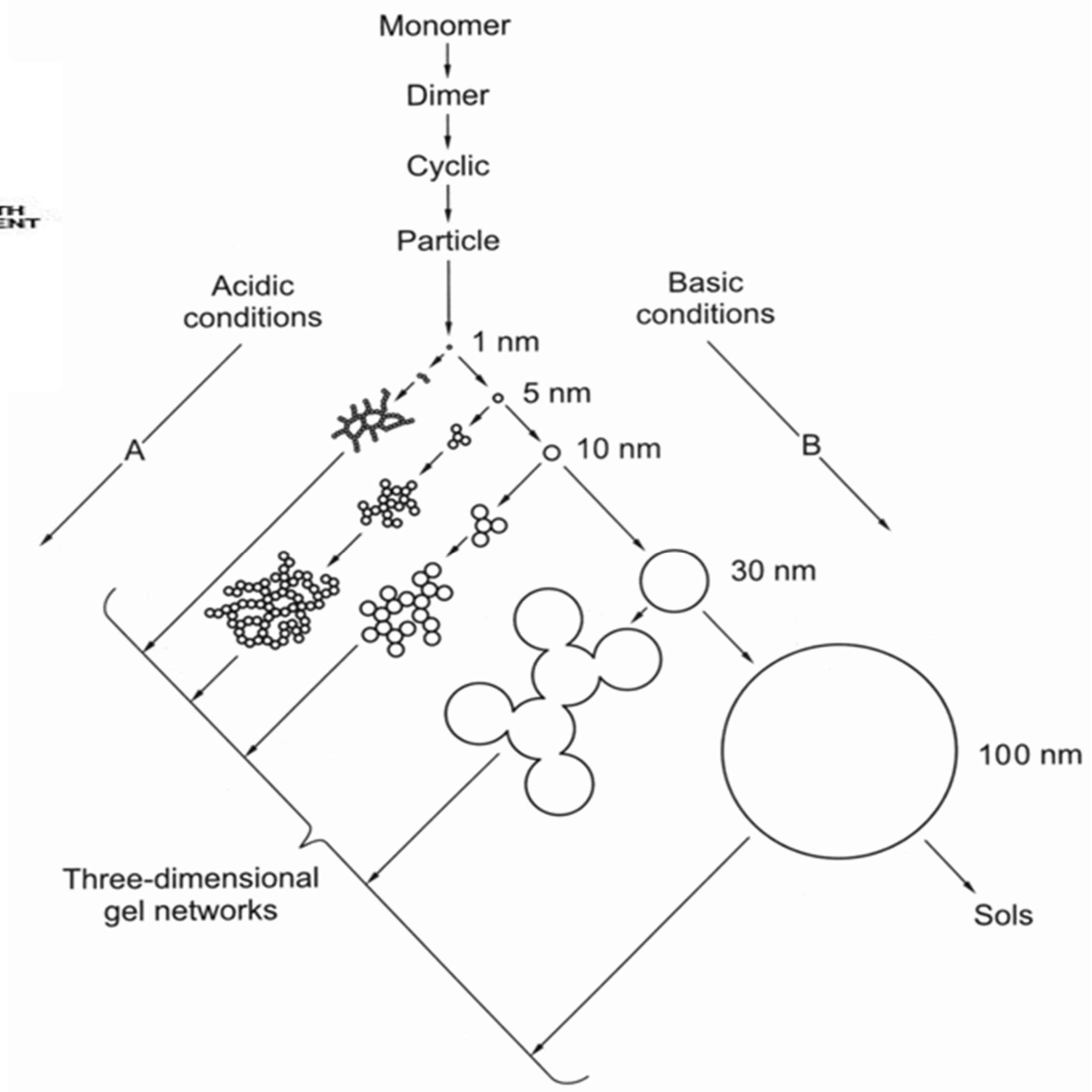


7. Relative and absolute concentrations of the reactants

It was found that the rate of hydrolysis and condensation was inversely proportional to the amount of ligand added to the solution.

Network formation





Two Competitive Mechanisms of Condensation

1. oxolation : formation of oxygen bridges : 氧橋合

$$\text{M-OH} + \text{XO-M} \rightarrow \text{M-O-M} + \text{XOH}$$

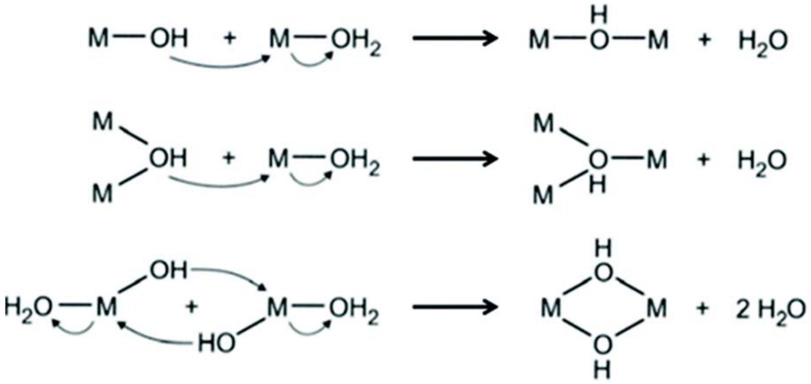
where X = H, generally when hydrolysis ratio h = $H_2O/M >> 2$
or X = R, when h = $H_2O/M < 2$

2. olation : formation of hydroxo bridges when the coordination of the metallic center is not fully satisfied (N - Z > 0) : 羥橋合

N: the coordination number
Z: the oxidation state

$$\text{M-OH} + \text{HO-M} \rightarrow \text{M-(OH)}_2\text{-M} \text{ where X = H or R}$$

The kinetics of olation are usually faster than those of oxolation.

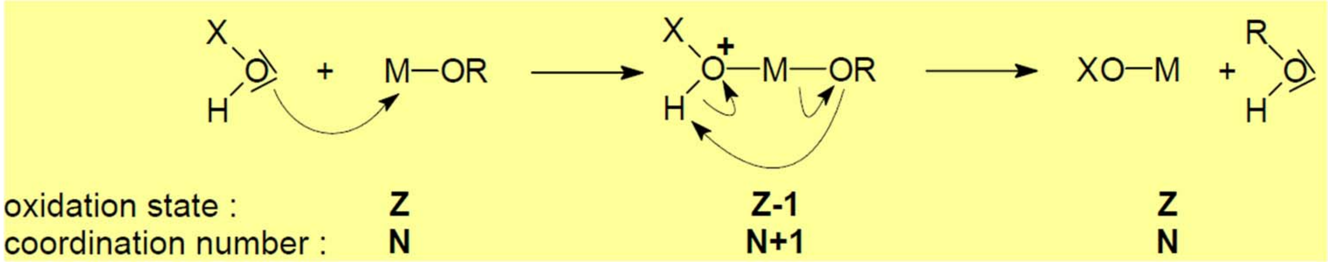


Other metal alkoxides

Chemical reactivity of metal alkoxides

M(OR)n + m XOH → [M(OR)n-m(OX)m] + m ROH

where X stands for H (hydrolysis),
M (condensation) or
L (complexation by an organic ligand L)



Consequently, the chemical reactivity of metal alkoxides toward nucleophilic reactions mainly depends on:

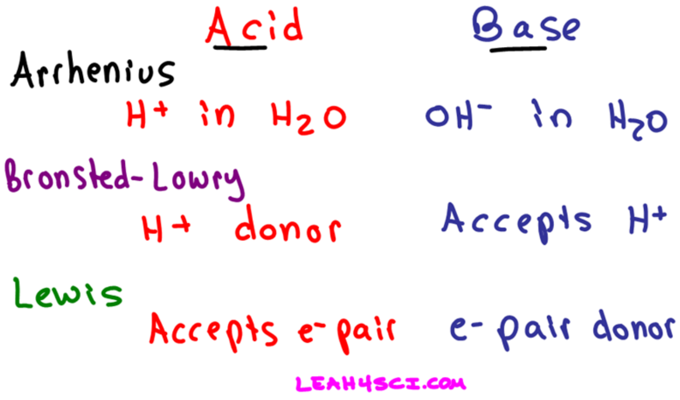
1. the strength of the nucleophile
2. the electrophilic character of the metal atom (an electronegativity-related property characterized by the positive charge on the metal atom)
3. its ability to increase its coordination number N: the degree of unsaturation of the metal coordination can be simply expressed by the difference N - Z, where N is the coordination number usually found in the oxide and Z is the oxidation state

More acidic than for Si

Metals may be used for formation of gels, almost as silicon. However, many metals will form oxides when increasing pH. The water molecules of the hydrated metal ions are more acidic than for Si, and hydrolyses will occur. The reactions are moved toward lower pH.

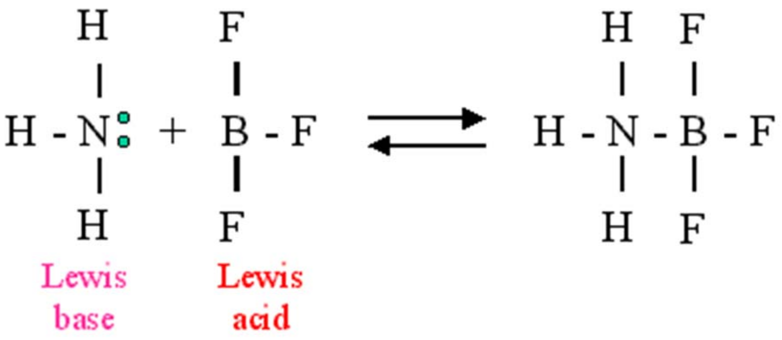
$pH < 3$	$pH > 3$	$pH >> 3$
$[Al(H_2O)_6]^{3+}$	$[Al(OH)_x(H_2O)_{6-x}]^{(3-x)+}$	Al-O-Al

其他金屬陰電性沒有比矽來得強，引此電子密度沒有像矽這麼多。也就是比較酸，比較容易接受電子對。因此比較容易受親核體的攻擊，闢需再比較酸的環境才能減緩氧化物的反應。



	Acid	Base
Arrhenius	H^+ in H_2O	OH^- in H_2O
Bronsted-Lowry	H^+ donor	Accepts H^+
Lewis	Accepts e- pair	e- pair donor

LEAH4SCI.COM



http://www.mikeblaber.org/oldwine/chm1046/notes/AcidBase/Lewis/Lewis.htm

Promote nucleophilic attack

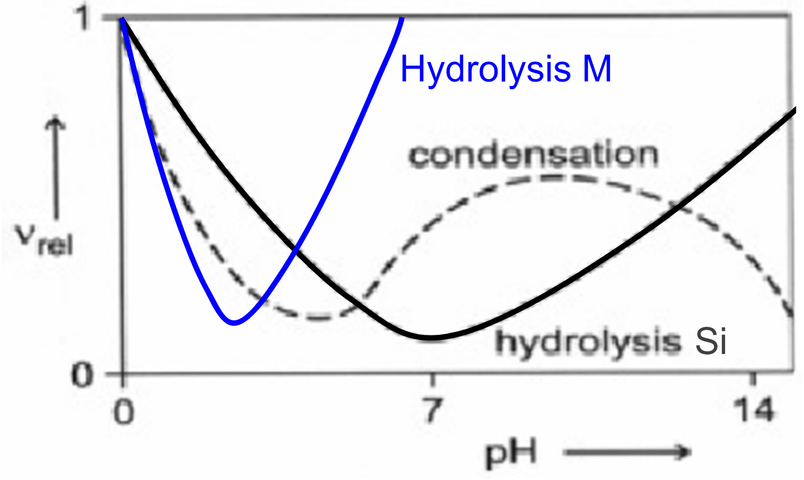
Differences between $Si(OR)_4$ and $M(OR)_x$

- Metal alkoxides are stronger Lewis acids and will promote nucleophilic attack.
- Hydrolysis of $Ti(OR)_4$ is up to 10^5 times faster than for the corresponding silicon alkoxide.
- Most metals have several stable coordination numbers or may easier expand the coordination sphere.

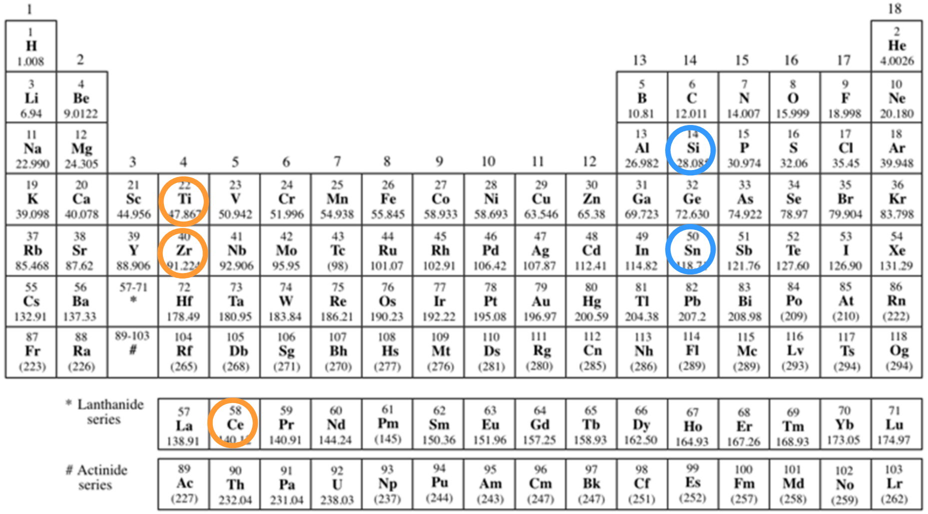
Hydrolysis, reactivity:

$$Si(O^iPr)_4 <<< Sn(O^iPr)_4, Ti(O^iPr)_4 < Zr(O^iPr)_4 < Ce(O^iPr)_4$$

When reacting with water many metal alkoxides form precipitates. While the alkoxy silanes needs catalysts, the reaction rates must be decreased for metal alkoxides.



The degree of unsaturation of the metal



alkoxides	χ	partial charge δ	ionic radius (Ǎ)	oxide	N	N - Z
$Si(OPr^i)_4$	1.74	+ 0.32	0.40	SiO_2	4	0
$Sn(OPr^i)_4$	1.89		0.60	SnO_2	6	2
$Ti(OPr^i)_4$	1.32	+ 0.60	0.64	TiO_2	6	2
$Zr(OPr^i)_4$	1.29	+ 0.64	0.87	ZrO_2	7	3
$Ce(OPr^i)_4$	1.17	+ 0.75	1.02	CeO_2	8	4

For similar alkoxy groups OR :

$Si(OR)_4 <<< Sn(OR)_4$

and $Ti(OR)_4 < Zr(OR)_4 < Ce(OR)_4$

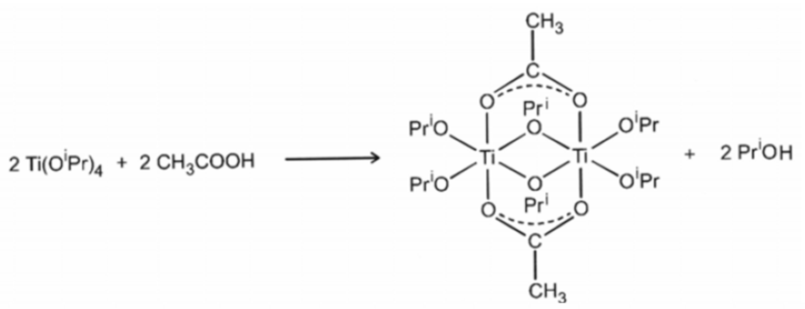
- silicon : relatively low electrophilicity and N - Z = 0 ; thus, silicon alkoxides are not very reactive : hydrolysis-condensation reaction rates must be increased by catalysts
- tin : higher electrophilic power and higher degree of unsaturation N - Z
- titanium, zirconium and cerium : lower electronegativity, but very high degree of unsaturation N - Z. Hydrolysis and condensation reactions of non-silicate metal alkoxides must be controlled (often reduced) by using chemical additives.

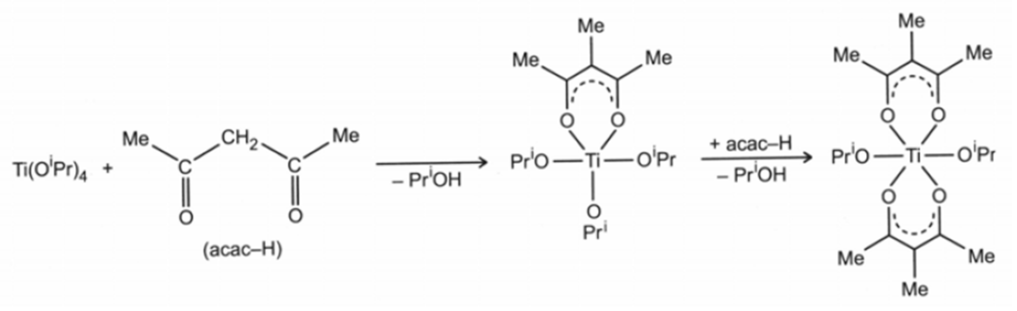
Reducing reactivity

- Metal alkoxides may be polymeric (silicon alkoxides tend to be monomeric).
  - ➢ Polymeric species react slower than monomeric.
- Small ligands result in faster reaction than large ligands
- Bidentate ligands also slow down the hydrolysis.

methyl ethyl propyl butyl pentyl hexyl

甲 乙 丙 丁 戊 己

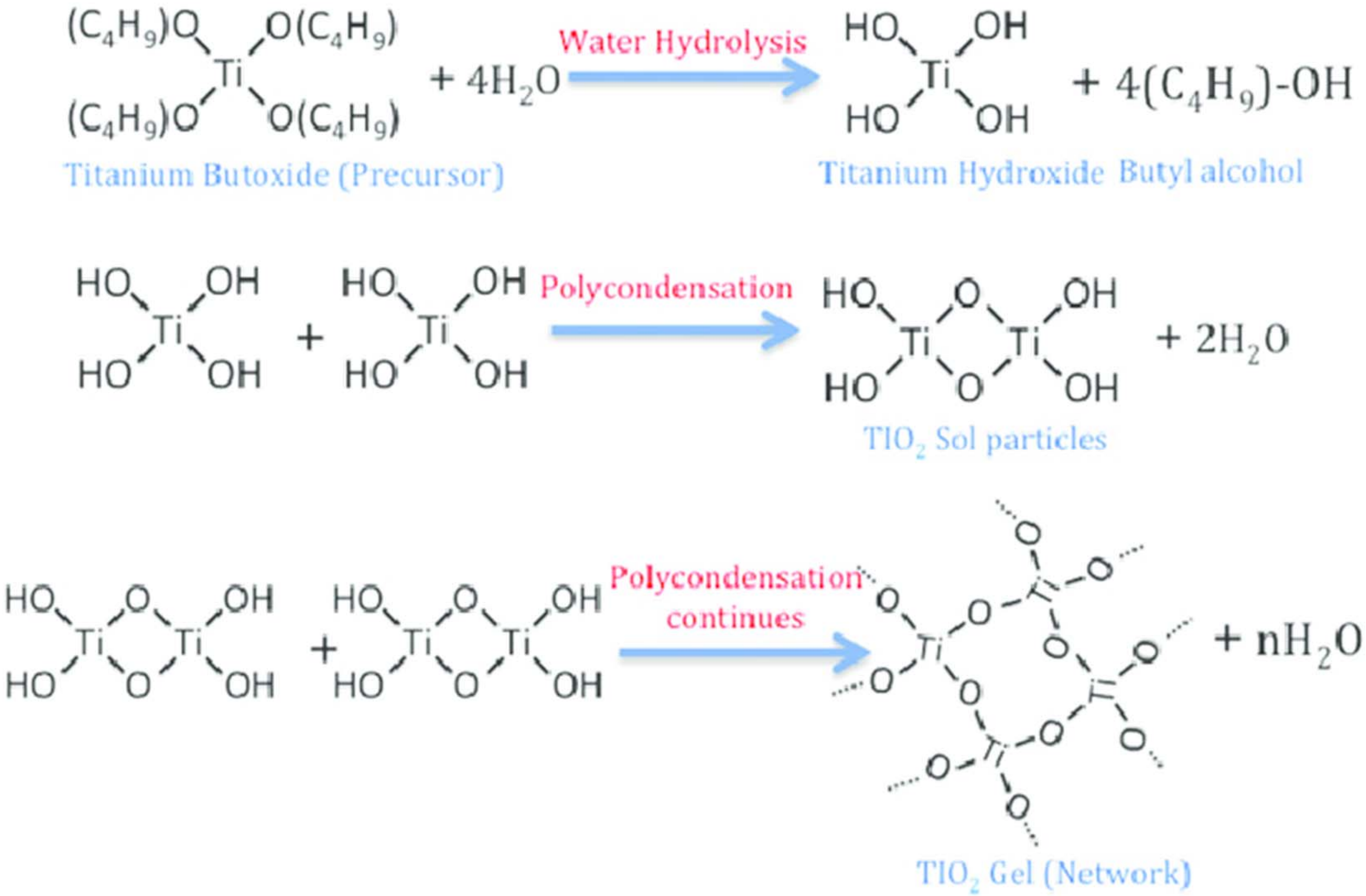




For Titanium alkoxide

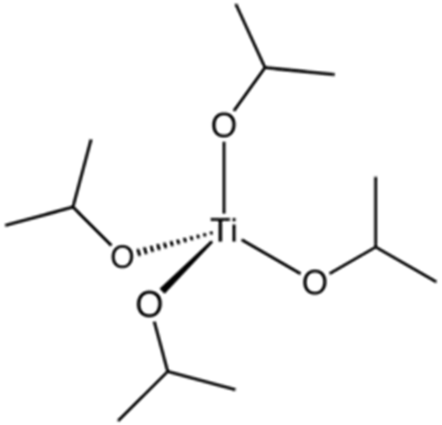
Sol-gel of Titanium alkoxide

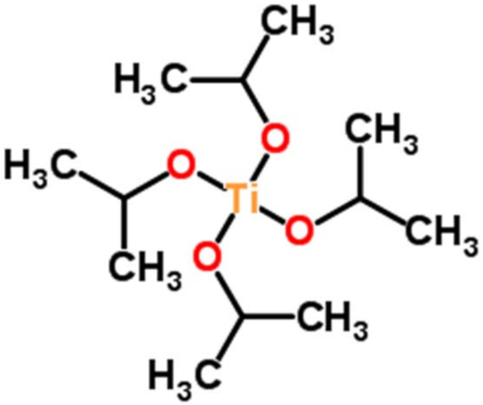
醇鹽



https://www.researchgate.net/figure/Schematic-representation-of-sol-gel-reaction-of-titanium-dioxide_fig1_265604410

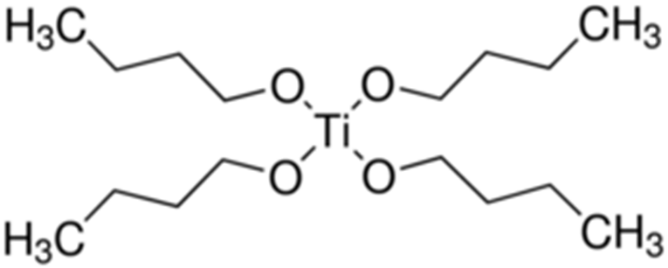
Formula of Titanium(IV) alkoxide



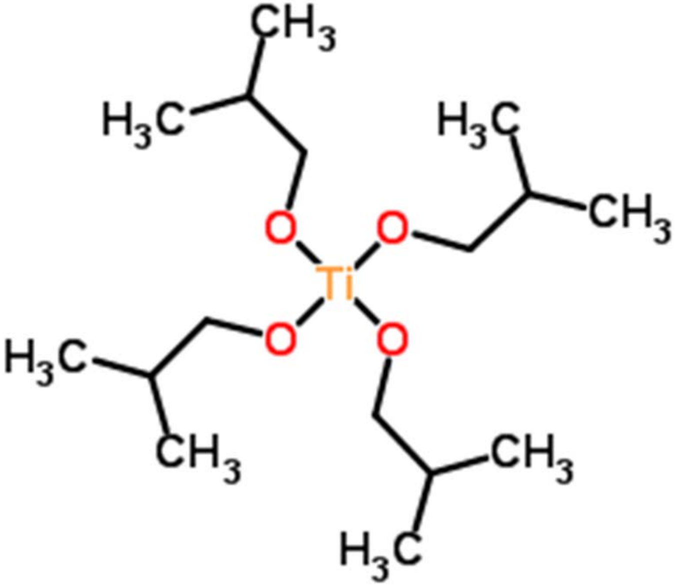


TTIP $Ti(O^{i}Pr)_4$

Titanium *tetra*-isopropoxide



Titanium(IV) n-butoxide

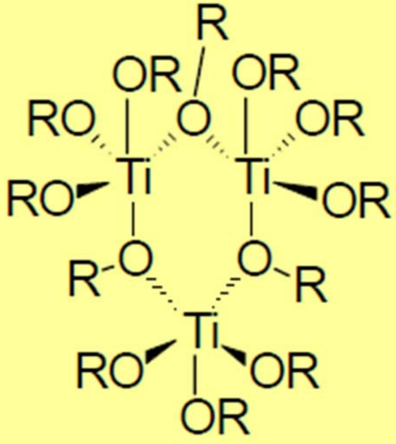


Titanium(4^{+}) tetrakis(2-methyl-1-propanolate)

Titanium butoxide

Molecular complexity

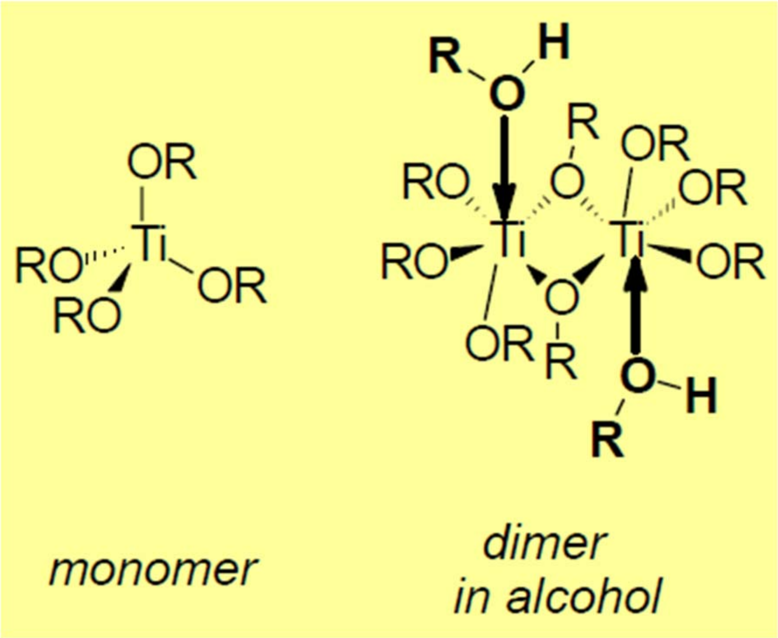
- the molecular complexity depends on concentration, temperature, solvent, oxidation state of the metal, steric hindrance of the alkoxy groups.
- the molecular complexity increases with the atomic size of the metal atom and decreases with increasing bulkiness of the OR groups.
- in non-polar solvents, the molecular complexity of alkoxides is higher than in their parent alcohol, and therefore their hydrolysis reaction is slower and leads to the formation of clear gels.



trimer
in pure benzene

High Reactivity

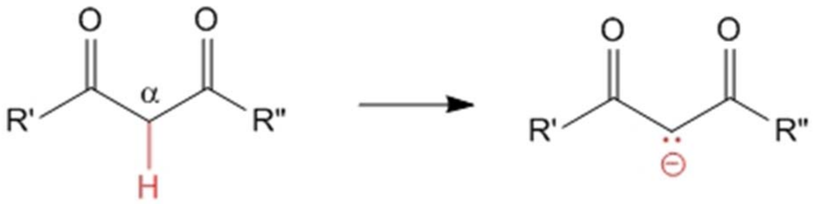
1. Very sensitive to moisture, they must handled with care under a dry atmosphere otherwise precipitation occurs.
2. Coordination expansion: self regulation of their reactivity by increasing the number of coordinating molecules using their vacant orbitals to accept electrons from nucleophilic ligands.



Control (reduction) of the reactivity with inhibitors

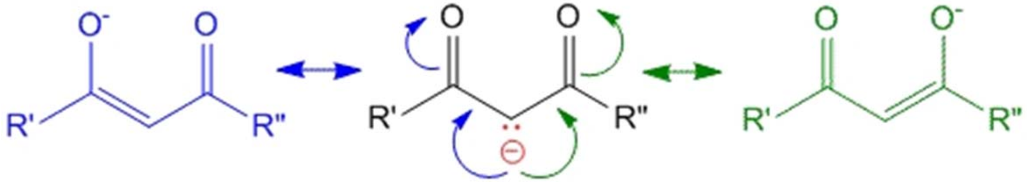
Control of the reactivity with inhibitors : inorganic acids, β- diketones, carboxylic acids or other complexing ligands which inhibit condensation reactions and therefore prevent precipitation, force the growth to a gel state or stop growth.

Ti–OR bonds will be replaced by the β-diketone and are no longer available for hydrolysis or further condensation.

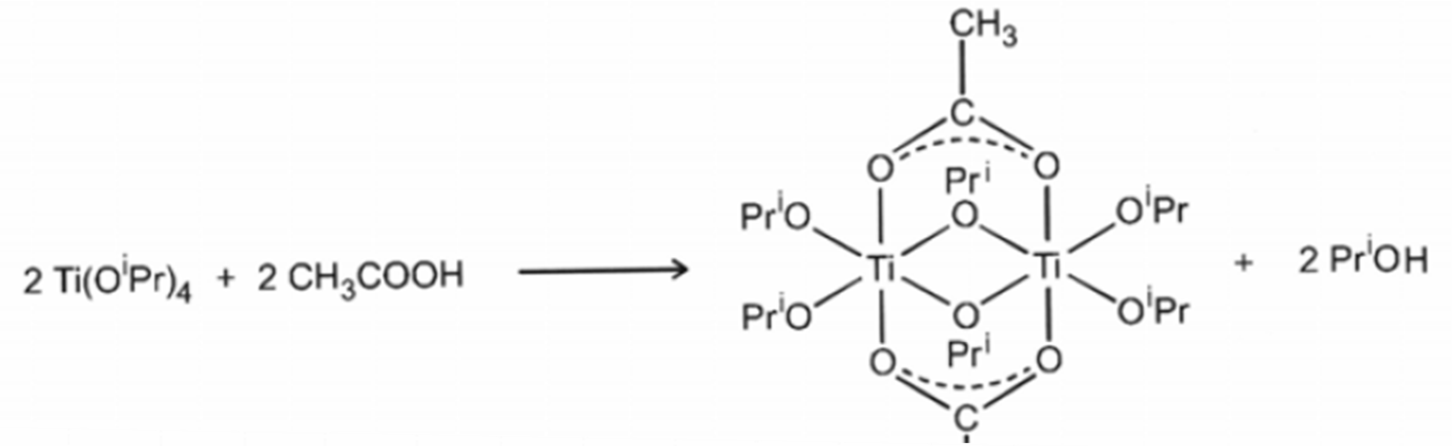


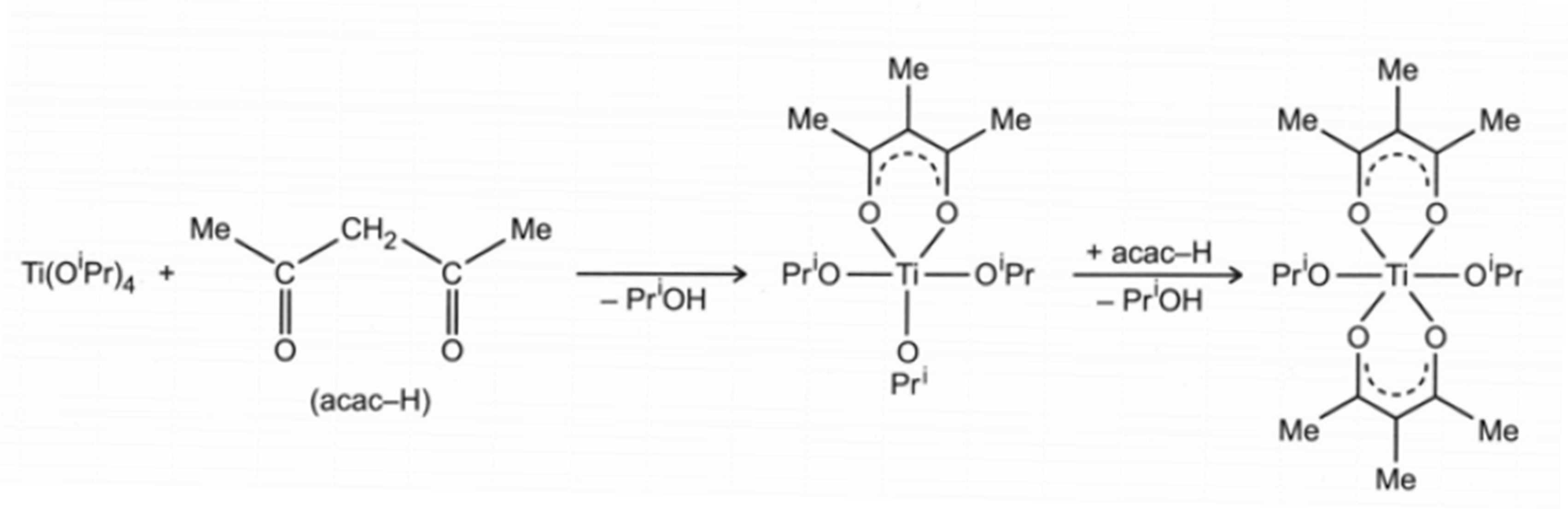
β-diketone α–hydrogen
pKa = 9

Reason -

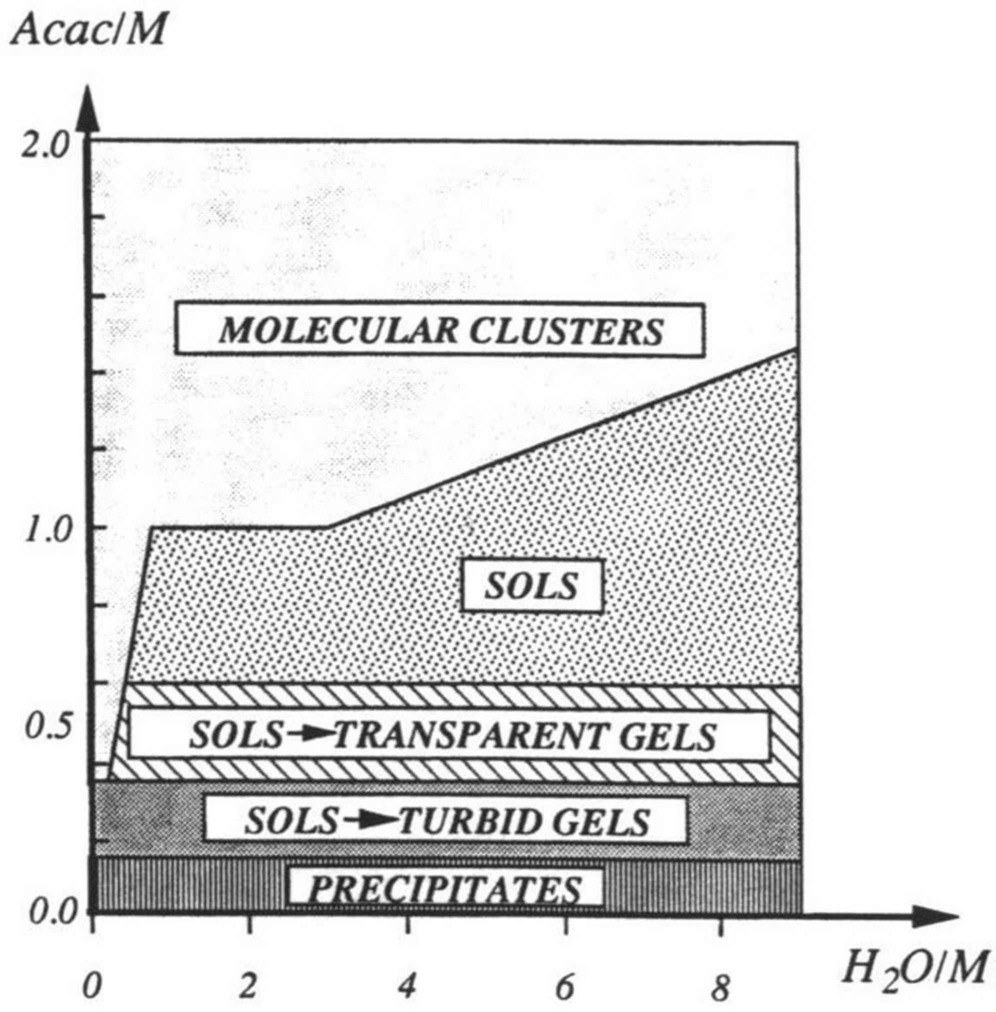


Beta-diketones have extremely acidic alpha-hydrogens (pKa = 9), mainly because the formed negative charge on the conjugate base can be distributed on both the ketone groups on either side.





ex : acetylacetone (AcacH) leads to less hydrolyzable M-Acac bonds.



State diagram describing the hydrolysiscondensation behavior of complexed tetravalent metal alkoxides

Aging

As the viscosity rapidly increase, the solvent is “trapped” inside the gel.
The structure may change considerably with time, depending on pH, temperature and solvent. The gel is still “alive”.

- The liquid phase still contains sol particles and agglomerates, which will continue to react, and will condense as the gel dries.
- The gel is originally flexible. Groups on neighbouring branches will condense, making the gel even more viscous. This will squeeze out the liquid from the interior of the gel, end shrinkage occur. This process will continue as long as there is flexibility in the gel.
- Hydrolysis and condensation are reversible processes, and material from thermodynamically unfavourable points will dissolve and precipitate at more favourable points.

(Note the similarity to the sintering process)

Forming with Aging

TEOS:Ethanol:Water
Molar Ratio 1 : 1 : 1

Add HCl
TEOS:HCl Molar Ratio 100:1
Hydrolyze for 45 min

$Y(NO_3)_3$: $Er(COOCH_3)_3$
4:1 or 200:1

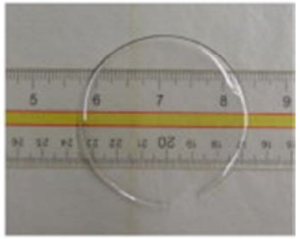
Add HNO_3
$(Y+Er):HNO_3$ 100:1
Dissolve in water for 45 min

Combine
Mix for 2½ hours

Pour into polystyrene petrie dishes
or polypropylene tubes

Gel for 2-7 days

Dry and age at 60ºC for 2 weeks



Heat treat to form densified samples:
Heat at 15ºC/h from room temperature to 250ºC, hold 10 hours;
Heat at 30ºC/h to 600ºC, hold for 10 hours;
Heat at 60ºC/h to 1000ºC, hold 10 hours.
Cool to room temperature at 60ºC/h.

Lisa Klein, Journal of Non-Crystalline Solids 354 (2008) 3567–3571

Drying

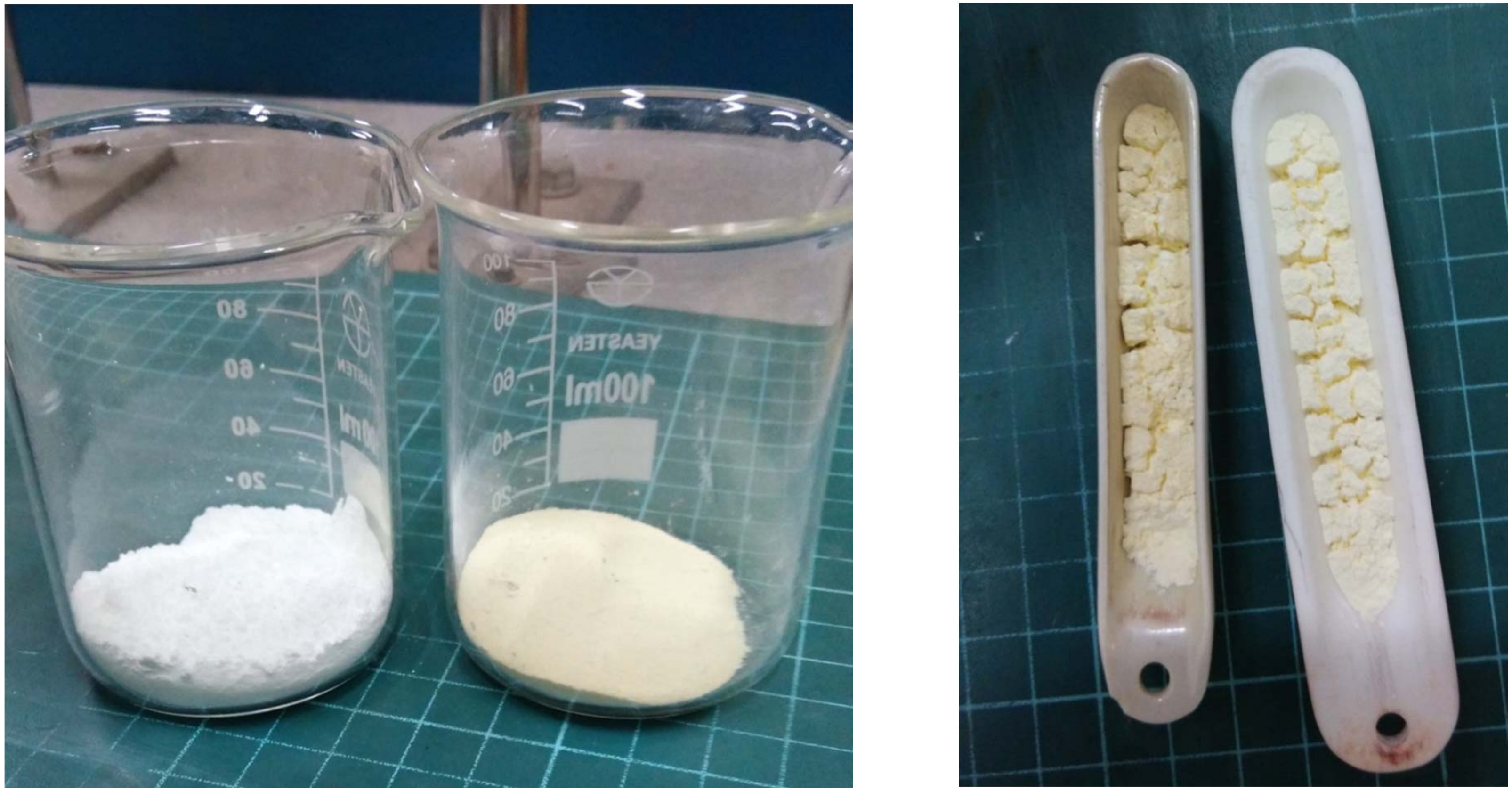
When the liquid is removed from the gel several things may happen. When the liquid in the gel is replaced by air, major changes to the network structure may occur.

If the structure is maintained, an aerogel is formed If the structure collapses, a xerogel is formed.

- Normal drying of the gel leads to structural collapse due capillary forces drawing the walls of the pores together, and reducing the pore size.
- OH groups on opposite sides may react and form new bonds by condensation.
- Cracking may occur when the tension in the gel is so large that it cannot shrink anymore.
- Gas will enter the pores with a thin film of liquid on the walls. This will evaporate and only isolated spaces with liquid are left.

Final Products

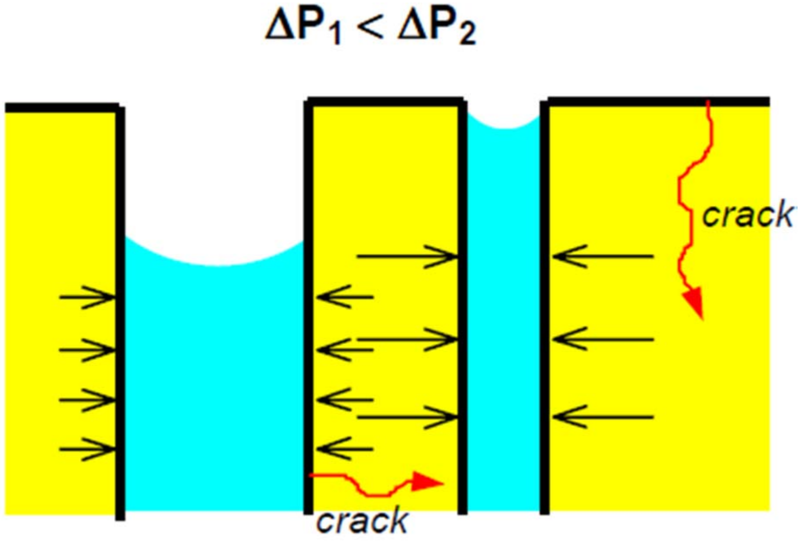
If **powdered ceramics** are desired, no special care need be exercised to prevent fragmentation. (This is what we usually do for photocatalyst synthesis)



Final Products

If **monoliths from colloidal gels** are desired, the drying procedures are largely determined by the need to minimize internal stresses associated with the volume changes on drying and the capillary forces in the gel pores :

$$\Delta P = 2\gamma\cos\theta/r$$



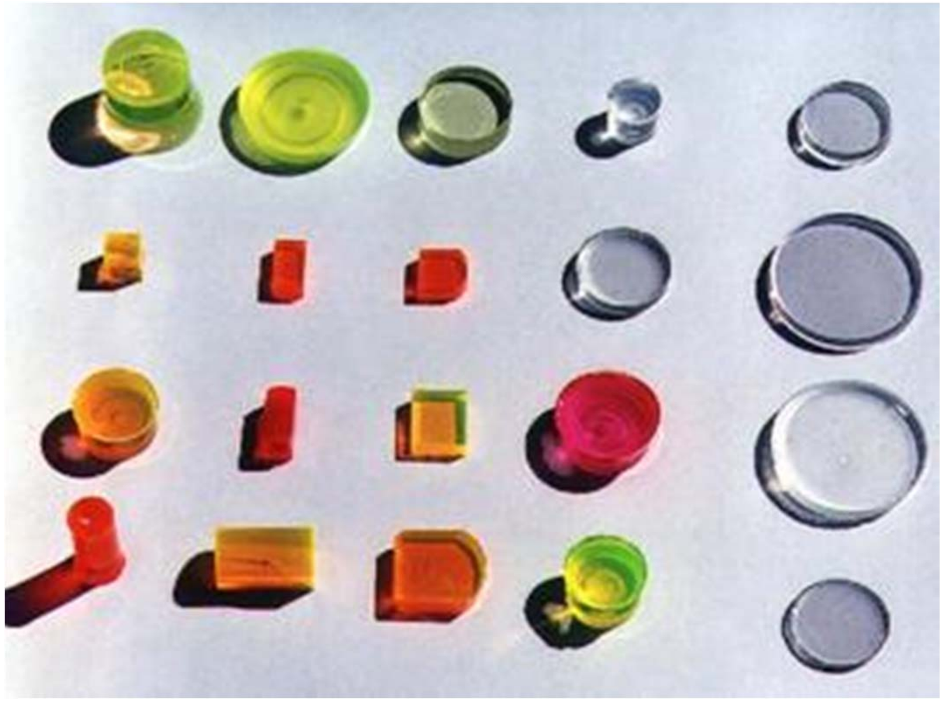
Final Products

If **monoliths from polymeric gels** (such as those obtained by the acid catalysis of silicon alkoxide at low water contents) are desired: the polymeric species are weakly crosslinked and as solvent is removed, they can deform readily and form a dense gel structure while polymer itself shrinks and forms more crosslinks

- ▶ dense dried gel with no large voids

Sol-gel Products after Drying

By using sol-gel technology we are synthesizing and studying the following materials:



- ANTIREFLECTING COATING
- ELECTROCHROMIC WINDOWS
- GASOCHROMIC WINDOWS
- HEAVY METAL AND pH SENSORS
- SEMICONDUCTOR QUANTUM DOTS AND NANORODS
- SOLID STATE TUNABLE LASERS
- VISUAL SENSING SYSTEM ASSISTING IN DRIVING PRECAUTION

T. Saraidarov, R. Reisfeld, A.Sashchiuk and E. Lifshitz, , Journal of Sol-Gel Science and Technology(2004)

http://chem.ch.huji.ac.il/~rena/PrezAct.html

Counteract the Capillary Forces

Excessive capillary forces can be reduced or eliminated by drying under **hypercritical conditions.**

Effected by heating the gel with methanol in an autoclave to temperatures and pressures exceeding the critical point of methanol (240°C/8.1MPa). A slow evacuation (3-4 hr) was followed by flushings with dry argon to eliminate the last traces of alcohol. This method produces dried gels with much larger pore volumes (aerogels: porosity of ~85 %, specific surface area of 250-600 $m^2 \cdot g^{-1}$ and pore size of 250-1250 Å) than conventional evaporative drying (xerogels : pore size ≤ 20 Å). Because of the absence of the liquid/vapor interfaces, no capillary forces arise to cause network collapse and shrinkage during drying.

Homework

Read 2 papers.

1. Sol-Gel processing of silica nanoparticles and their applications

https://www.sciencedirect.com/science/article/pii/S0001868614002802

2. Sol–gel synthesis of erbium-doped yttrium silicate glass–ceramics

https://www.sciencedirect.com/science/article/pii/S0022309308001609