Methods of preparation of colloids

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Classification of colloids (Based on interaction or affinity of phases)

- Lyophilic Colloids

DM DP

- Lyophobic Colloids

DM DP

Property	Lyophilic	Lyophobic
Nature	Reversible	Irreversible
Preparation	Shaking or warming DP with DM	Special method used
Stability	Very stable	Unstable, easily coagulated on addition of electrolytes
Solvation	Heavily solvated	Particles not solvated
Migration in electric field	May or may not	Migrate either towards Anode or cathode
E.g.	Starch, gums, proteins, gelatin, etc	We will discuss…..

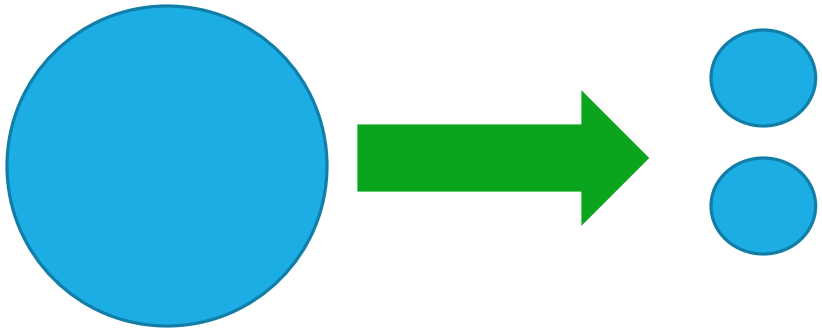
Preparation of Lyophilic sols

- In this method, the dispersed phase is directly added to dispersion medium either in cold or on warming.
- Colloidal solution of starch, gelatin, glue in water
- Solution of colloidal electrolytes ,e.g. soaps, dye

Preparation of Lyophobic sols

❑ Top-down approach

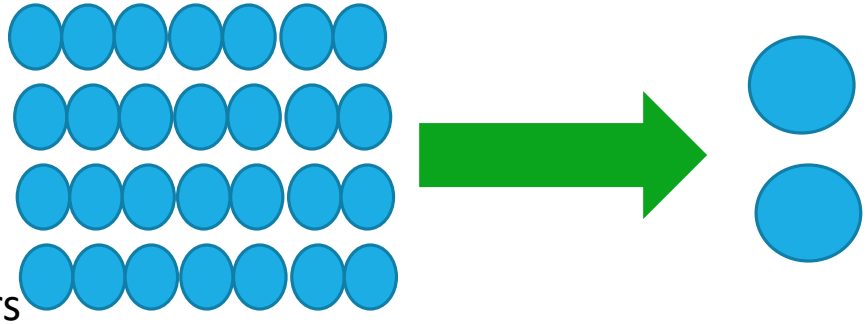
- ❖ Crushing or Grinding
- ❖ Electrical disintegration
- ❖ Laser Ablation
- ❖ Normal boundary motion
- ❖ Tangential boundary motion
- ❖ Electrical emulsification or atomization
- ❖ Lithography



✓ **Dispersion methods**

❑ Bottom-down approach

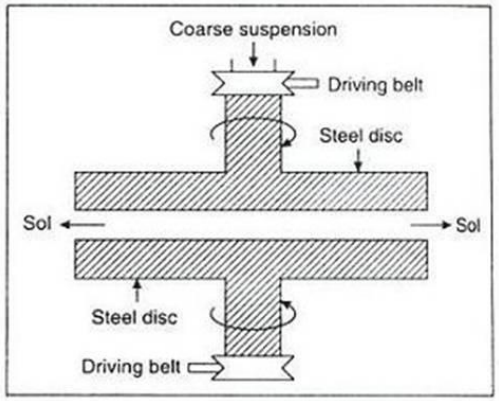
- ❖ Controlled precipitation
- ❖ Reduction of metal complexes
- ❖ Sol-gel method
- ❖ Pyrolysis of metal-organic precursors
- ❖ Emulsion, dispersion, suspension polymerization
- ❖ Amphiphile self-assembly in solution
- ❖ Physical or chemical vapor deposition
- ❖ Synthesis and growth within confined templates



✓ **Condensation methods**

Top-down approach

- Mechanical comminution (mechanical disintegration)



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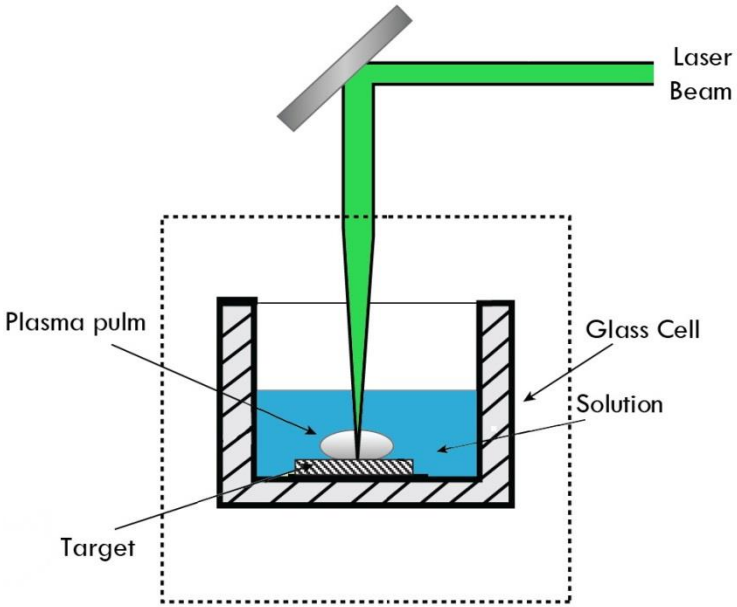
- Solid material finely ground first
- Mixed with DM to get coarse suspension
- Suspension introduced into colloidal mill
- Two heavy metal discs separated by a small gap
- R.p.m -7 to 8000
- The sols results due to large shearing effect
- A stabilizer is added
- E.g. Colloidal graphite (lubricant), printing ink

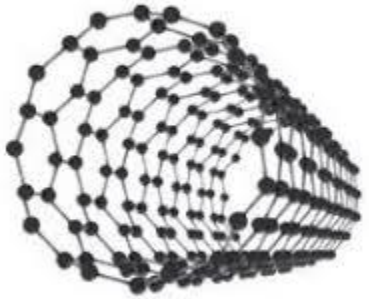
Graphite powder

Lamp black

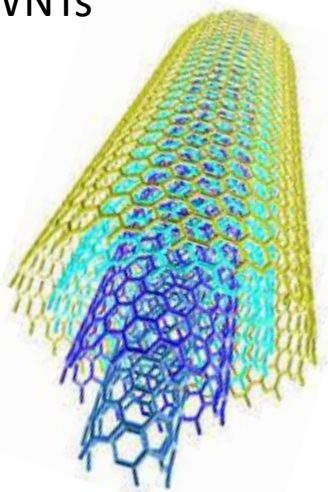
Top-down approach

- Laser ablation





SWNTs

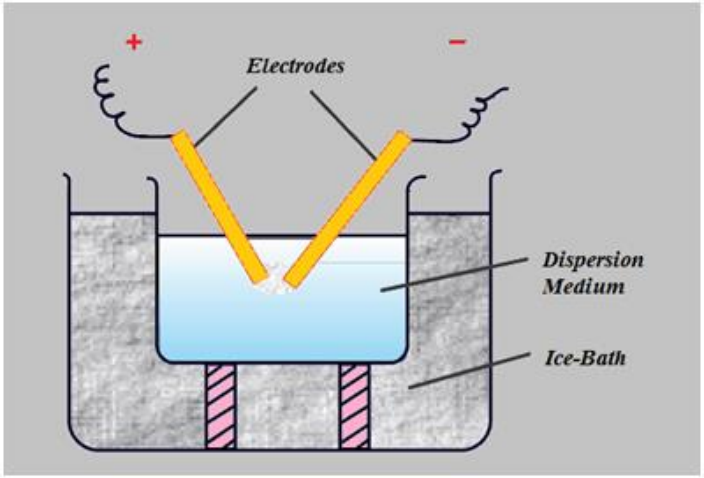


MWCTs

- High power laser is directed at the surface of a bulk material
- They produce ejecta, that are collected , sorted and analysed
- Single or multi-walled carbon nano tubes (SWNTs or MWCTs), other nanopowders

Top-down approach

- Electrical disintegration (Bredig's arc method)

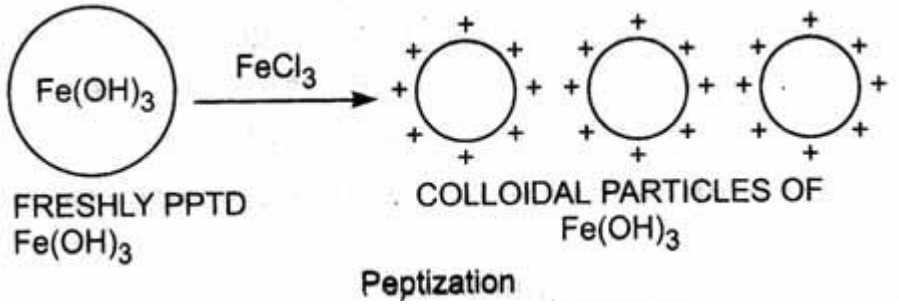


- This method is suitable for preparing colloidal solutions of metals like Au, Ag, Pt
- When a high voltage is applied, an electrical arc is struck between the tips of electrodes. The heat produced by the arc vaporizes the metal which condenses under cold water.
- NaOH or KOH in trace amount is used as stabilizer.

Top-down approach

- Peptization or chemical dispersion

  - Redispersion of freshly prepared precipitate into the sol by adding an electrolyte containing common ion.
  - The peptization is due to the preferential adsorption of one of the ions of the electrolyte on the precipitate.

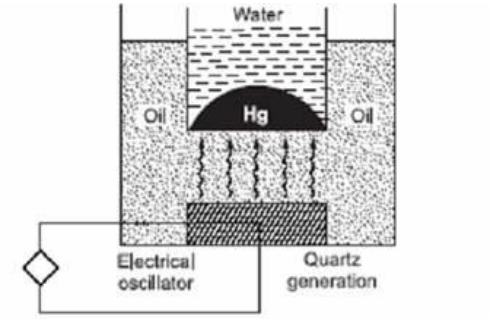


  - Freshly prepared AgCl can be converted into a colloidal solution by a small amount of HCl

  - More examples?

Top-down approach

- Dispersion of a liquid into a gas or another liquid
  - Shaking
  - Ultrasonic dispersion



Ultrasonic dispersion Process

Ultrasonic dispersion

- High intensity ultrasonic waves are used for this purpose
- The ultrasonic waves generated propagate through the DM and results in high pressure (compression) and low pressure (rarefaction) cycles.
- Mechanical stress produce Ultrasonic cavitation in liquids
- High speed liquid jets forms and press the liquid at high pressure between the particles and separate from each other.
- Substances like oil, mercury, sulphur, sulphides and oxides of metal can be dispersed by this method

Bottom-up approach

- Oxidation method

$H_2S + O_2 \longrightarrow H_2S + 2S$ (colloidal)

$H_2S + SO_2 \longrightarrow 2H_2O + 3S$ (colloidal)

- Reduction method
  - A number of metals like Ag, Au, Pt, Hg and Pb can be obtained in colloidal state by reduction of their salt solutions using suitable reducing agents (Hydrogen sulphide, tannic acid, stannous chloride, etc.)

$2AuCl_3 + 3\ SnCl_2 \longrightarrow 3SnCl_4 + 2\ Au$(colloidal)

$AgNO_3$ + tannic acid $\longrightarrow$ Ag sol (colloidal)

- Hydrolysis method
  - A colloidal solution of ferric hydroxide is obtained by hydrolysis of $FeCl_3$

$FeCl_3 + 3H_2O \longrightarrow Fe(OH)_3 + 3HCl$

Bottom-up approach

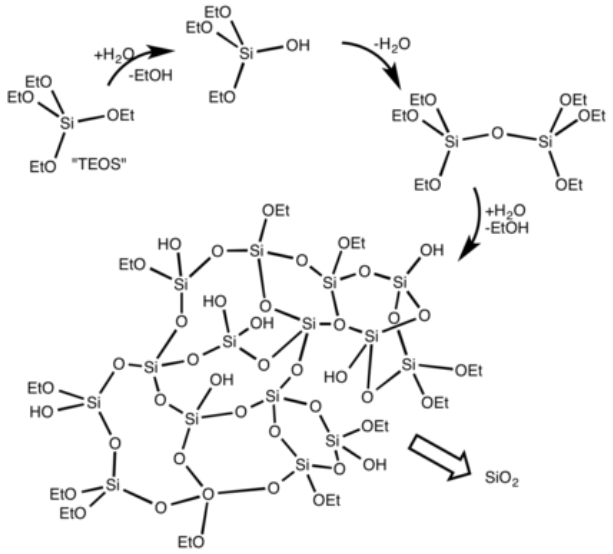
- Stober process (sol-gel approach)

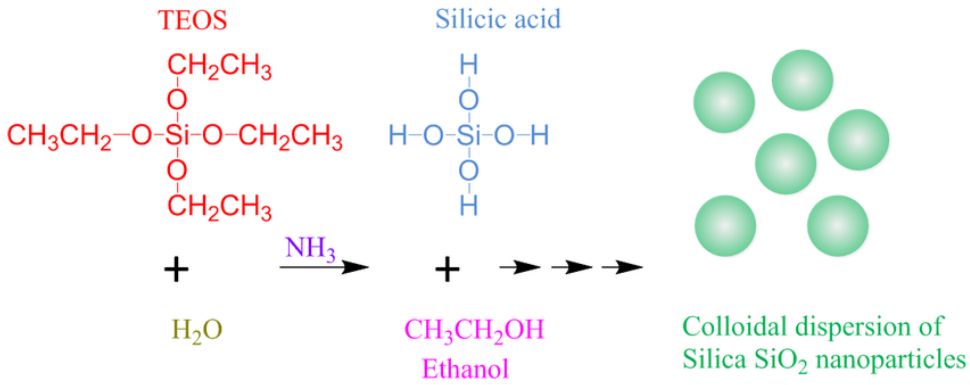
- The Stöber process is a sol-gel approach to preparing monodisperse (uniform) spherical silica (SiO_2) materials
- Silica precursor tetraethyl orthosilicate ($Si(OEt)_4$, TEOS) is hydrolyzed in alcohol (typically methanol or ethanol) in the presence of ammonia as a catalyst

$$Si(OEt)_4 + H_2O \longrightarrow Si(OEt)_3(OH) + EtOH$$

$$Si(OEt)_4 + 4H_2O \rightarrow Si(OH)_4 + 4\ EtOH$$

$$Si(OH)_4 \rightarrow SiO_2 + 2\ H_2O$$

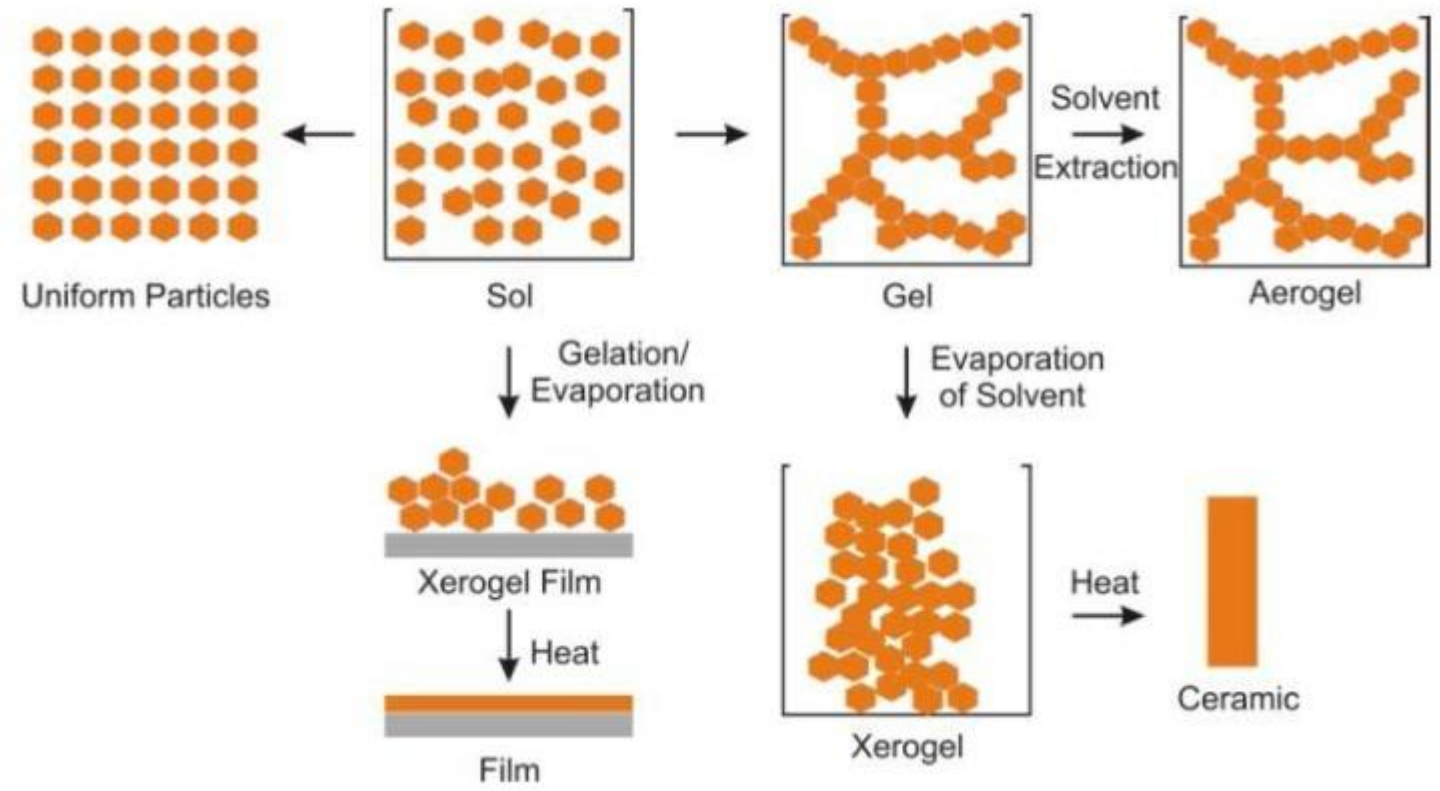




https://doi.org/10.4191/kcers.2017.54.1.10

Bottom-up approach

- Stober process (sol-gel approach)



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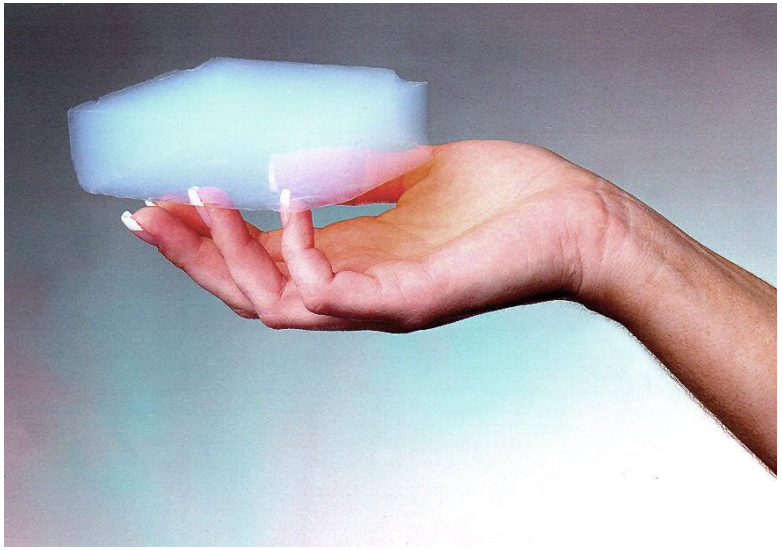
Bottom-up approach

- Stober process (sol-gel approach)

Silica gel is a commonly used desiccant as beads packed in a permeable bag



A block of an aerogel, a "solid blue smoke", which feels like very light-weight styrofoam to the touch

This NASA silica aerogel is a thermal insulator that is sufficiently powerful to protect crayons from a Bunsen burner flame.

Bottom-up approach

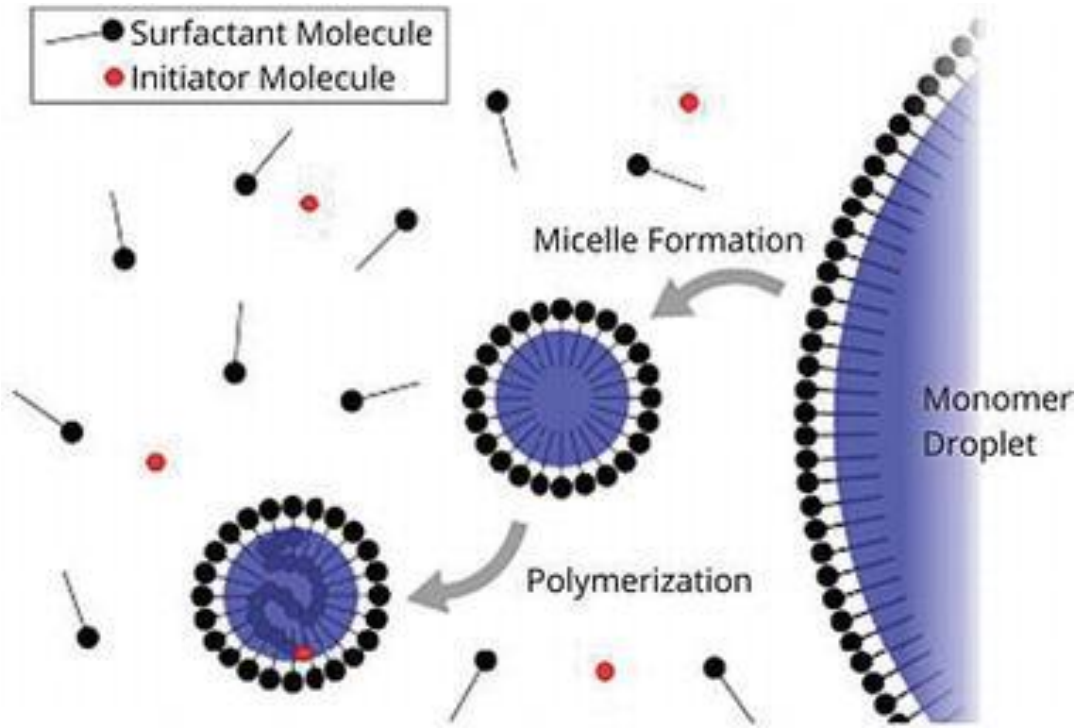
- Pyrolysis
  - Cadmium selenide may be produced by reacting dimethyl cadmium with tri-n-octylphosphine selenide at 300 ^{o}C in anhydrous solution of tri-n-octylphosphine and tri-n-octylphosphine oxide

$$Cd\ (CH_3)_2 + (C8H17)_3PSe \longrightarrow CdSe\ nanocrystals$$

- Emulsion polymerization
  - The main components of emulsion polymerization media involve monomer(s), dispersing medium, emulsifier, and water-soluble initiator
  - The dispersion medium is water in which hydrophobic monomers is emulsified by surface-active agents (surfactant)
  - When surfactant concentration exceeds critical micelle concentration (CMC) it aggregate in the form of spherical micelles, so surface tension at the surface decrease, as a result hydrophobic monomers enter in to the vicinity of micelle and reaction continue until all monomer droplets are exhausted and micelle containing monomers increase in size
  - Water-soluble initiators enter into the micelle where free radical propagation start
  - As polymerization continue inside micelle, the micelle grow by monomer addition from monomer droplets outside and latex are formed

Bottom-up approach

- Emulsion polymerization



Schematic representation of emulsion polymerization

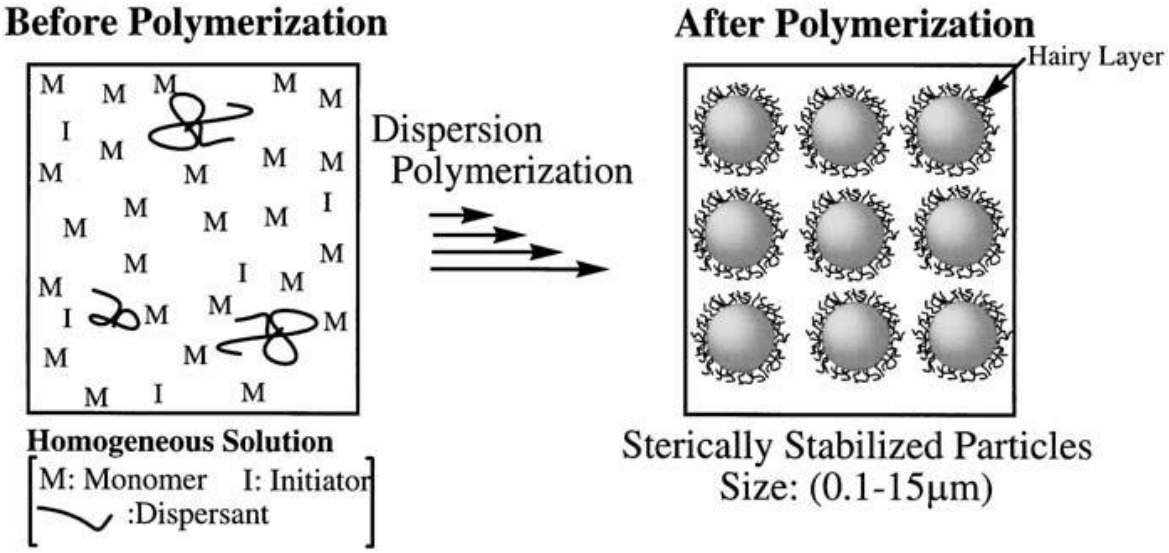
https://www.intechopen.com/books/recent-research-in-polymerization/emulsion-polymerization-mechanism

IUPAC definition

Emulsion polymerization: Polymerization whereby monomer(s), initiator, dispersion medium, and possibly colloid stabilizer constitute initially an inhomogeneous system resulting in particles of colloidal dimensions containing the formed polymer

Bottom-up approach

- Dispersion polymerization and suspension polymerization



- Double decomposition method

$$As_2O_3 + 3H_2S \xrightarrow{double\ decomposition} As_2S_3\ (sol) + 3H_2O$$

- Exchange solvent method

  - Solution of sulphur or phosphorous prepared in alcohol is poured into water, a colloidal solution of S or P is obtained. Why?