Lecture 5

Nanoparticles Synthesis Techniques

Synthesis

Several key issues of nanoparticle synthesis are: uniformity of particle size, size control, crystal structure, shape – control and alignment for various device applications:

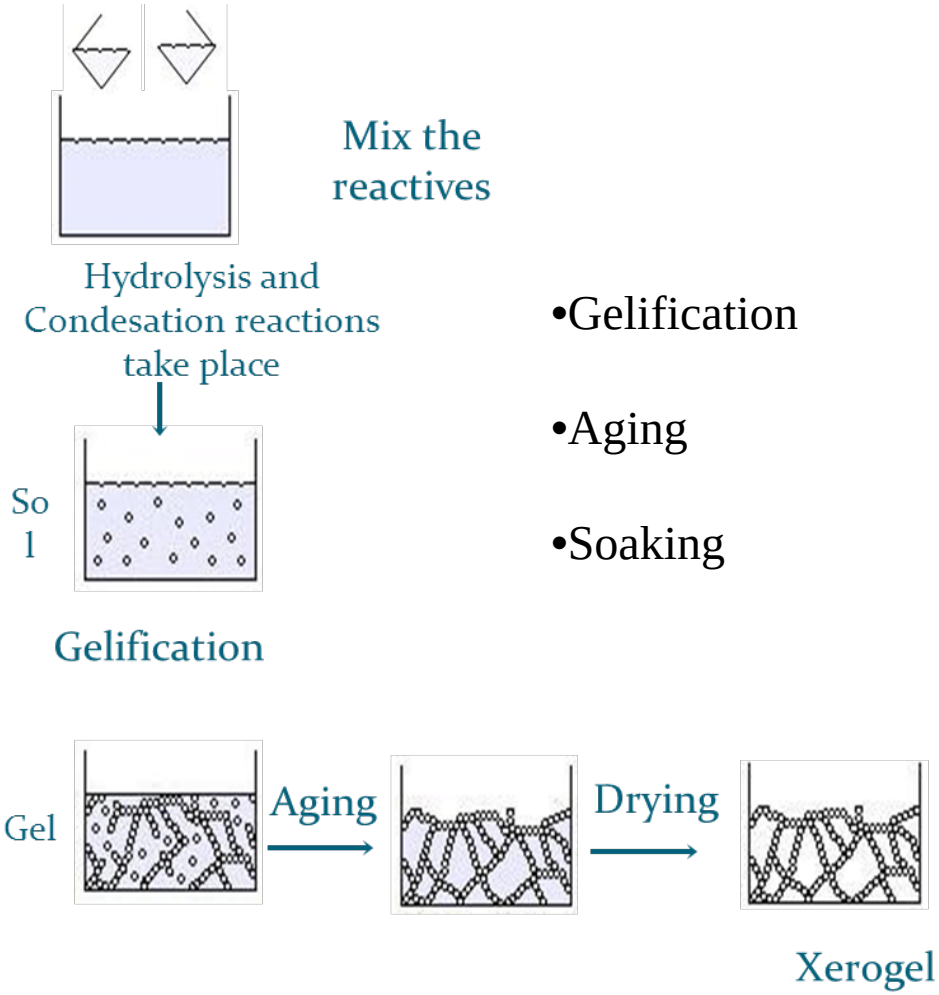
- The synthesis of discrete magnetic nanoparticles with sizes ranging from 2 to 20 nm is of significant importance, because of their applications in multi-terabit magnetic storage devices.

- The unique magnetic property of the nanoparticles arises mainly due to the reduced sizes of isolated nanoparticles where the contributions from inter particle interactions are negligible.

Chemical Synthesis

- Sol-gel
- Two-phase method (Microemulsion)
- Co-precipitation
- Polyols method
- Hydrothermal reaction
- Sonolysis
- Microwave irradiation

Sol-gel technique

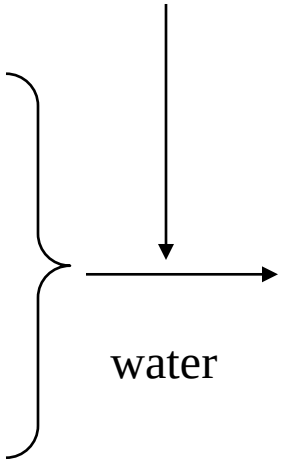


- •Gelification
- •Aging
- •Soaking

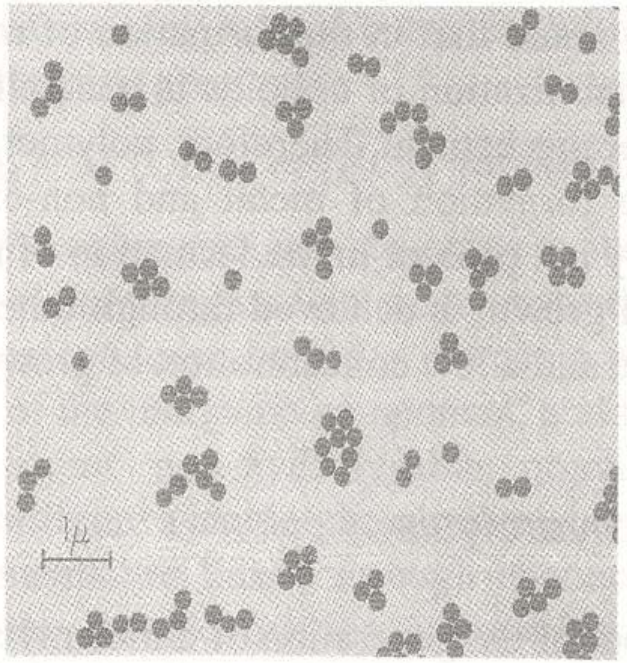
- Hydroxylation/ Hydrolysis
- Condensation
- Isothermal treatment.
- Obtain pure , size controlled stable and monodispersed nanoparticles ranges 20-200 nm.
- Iron oxide –silica gel composites are 2-3 times more reactive than conventional iron oxide.
- Contamination from by-products expected, product needs to be post treated.

Sol-gel example: silica

- Precursors:
  - silicone alkoxides/ oxides with different alkyl ligand sizes
- Nucleating agent/ catalyst:
  - ammonia
- Solvent:
  - Water/ various alcohols



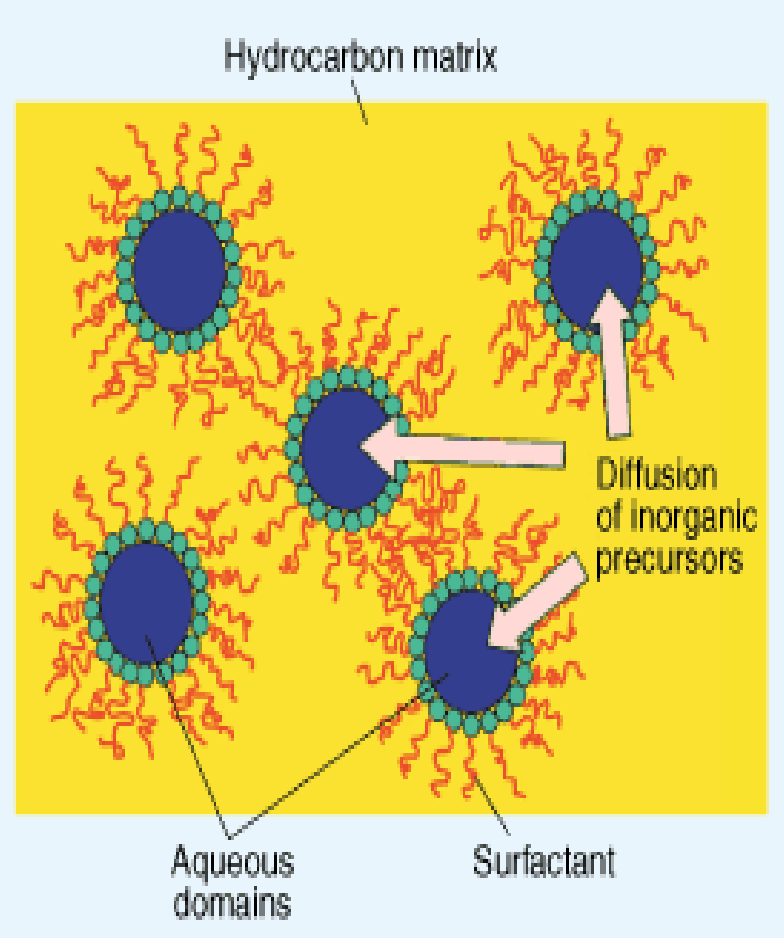
Vigorous stirring



Sol-gel products

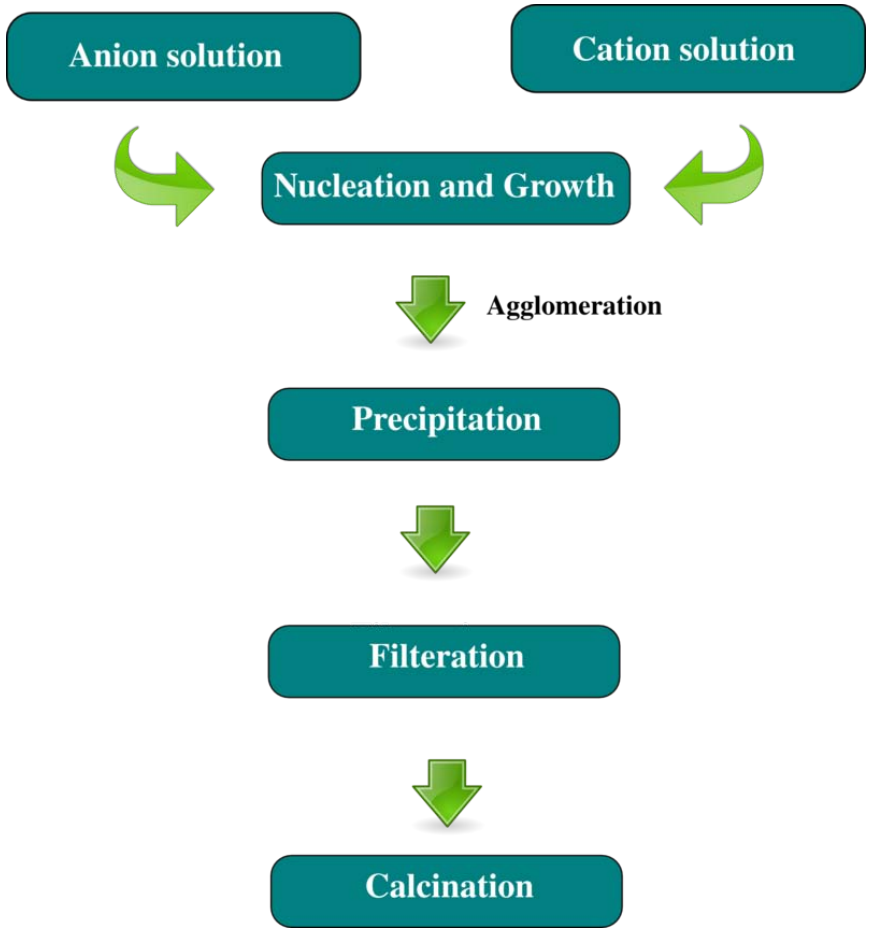
- Monodispersed (uniform size) nanoparticles
  - temporal nucleation followed by diffusion-controlled growth
  - size = f(concentration, aging time)
  - colloid stabilization: not by polymer steric barrier, by electrostatic double layer
  - complex oxides, organic-inorganic hybrids, biomaterials

Two-phase method(Microemulsion)



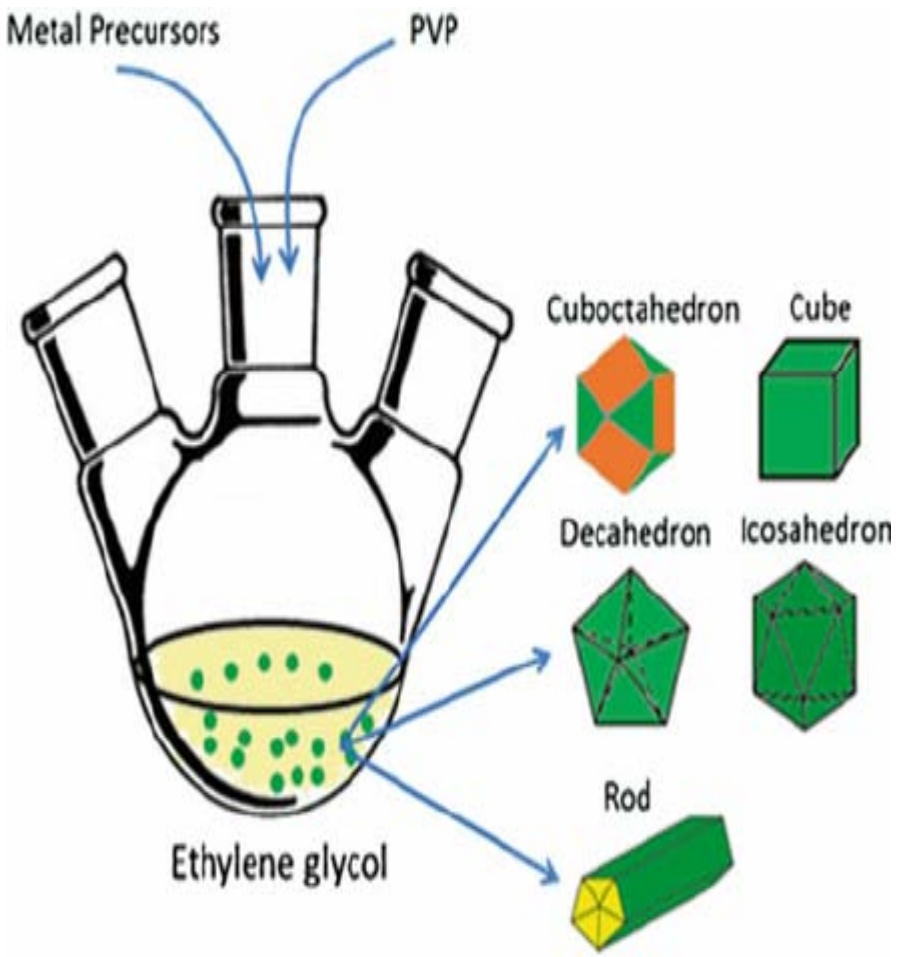
- Water in oil (w/o) emulsions
- Mono-dispersed nanoparticles
- Very good shape and size control
- 4-15 nm size with narrow size distribution obtained
- Surfactants are not easy to handle , low product yield obtained

Co-precipitation



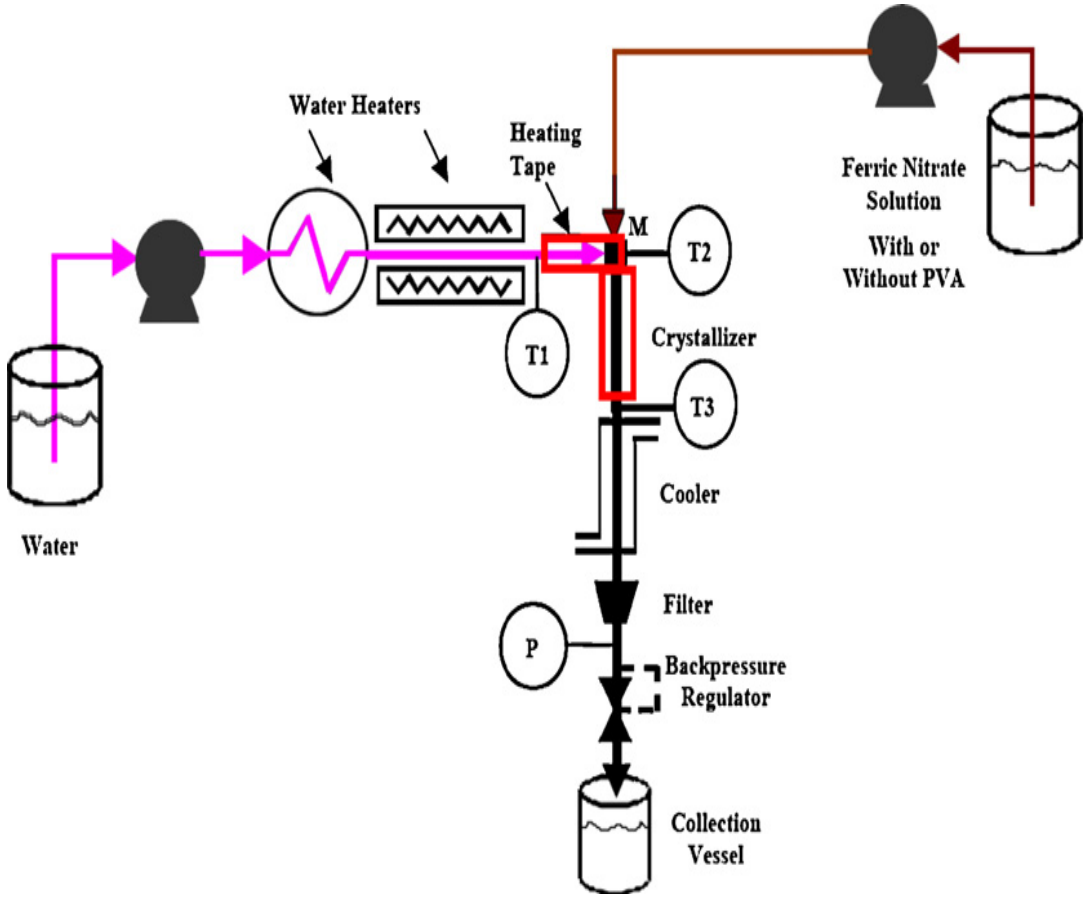
- Reduction by base
- By controlling pH and concentration of precursors, mean size of nanoparticles can be controlled from 2 -15 nm
- Ionic surfactants or protein coatings are used to avoid aggregation
- Particles obtained has broad size distribution

Polyols method



- Very promising technique for well defined shapes and size controlled nanoparticles for biomedical applications.
- Nucleation and growth process are completely separated, uniform nanoparticles are obtained with size ranges 10-90 nm.
- A polyol is an alcohol containing multiple hydroxyl groups.
- Types of polyols used, salts and their concentration, hydroxyl ion and reaction temperature are some important factors to control.

Hydrothermal reaction methods

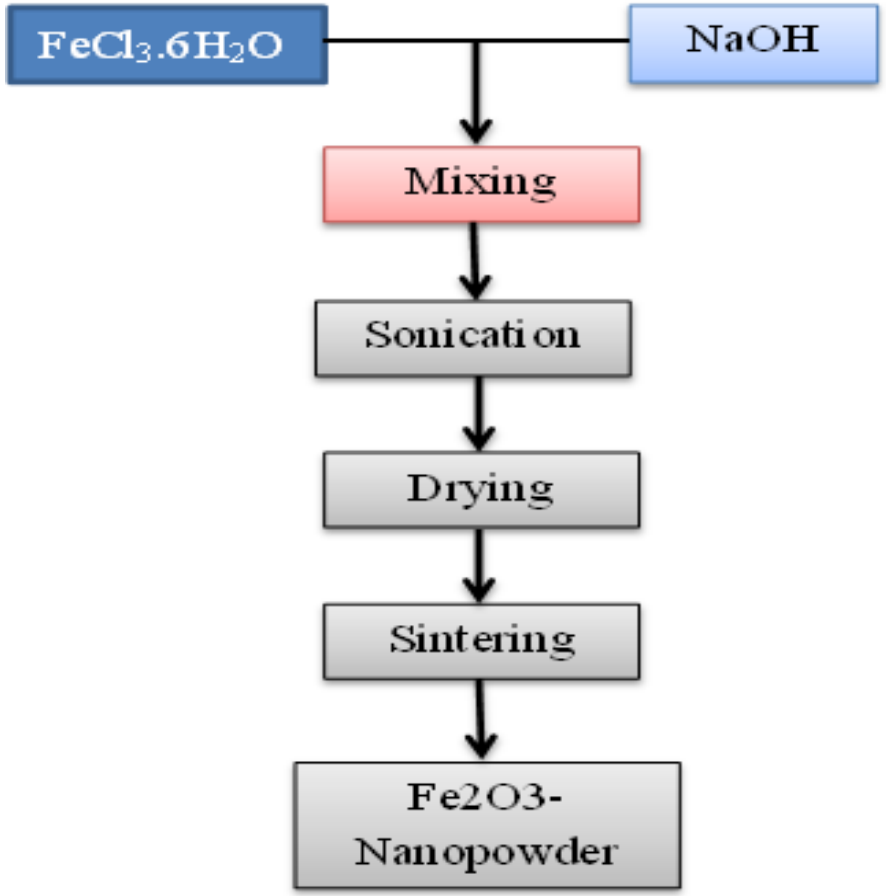


- High pressure ~ 2000 psi
- High temperature ~ 200 ^{o}C
- Monodispersed particles are produced at short residence time in aqueous media, obtained in a size ranges 10-50 nm
- Easy to scale up method, additional post-processing steps are required to achieve the engineered particle surfaces

Schematic diagram of the apparatus used for hydrothermal method

Sonolysis

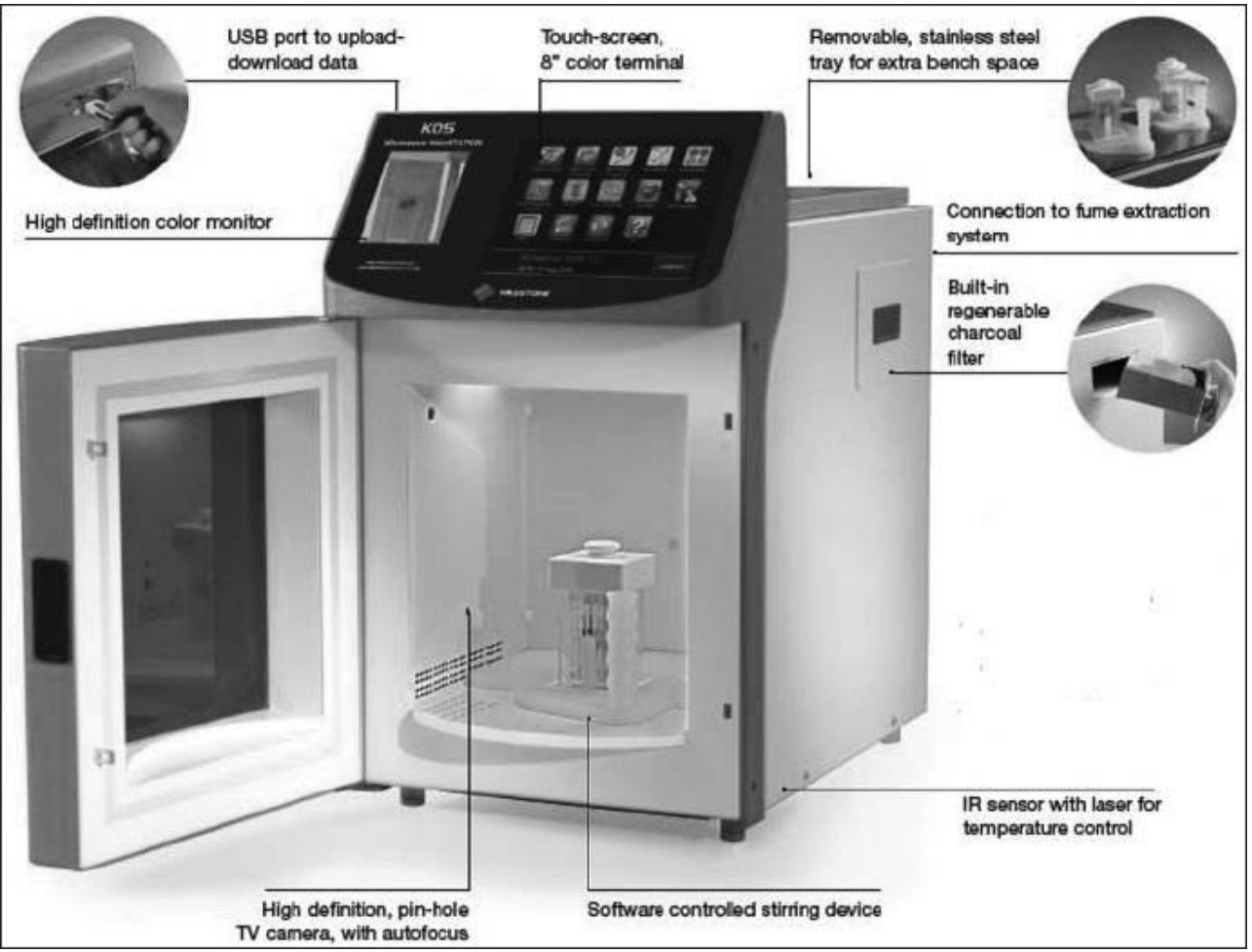
- Limit the growth, by forming cavitation in an aqueous media.
- Cavitation can generate temperature around 5000 ^{o}C, and pressure of over 1800 KPa
- Nanoparticles obtained from sizes range 20-40 nm
- Expensive equipments and very high temperature is required



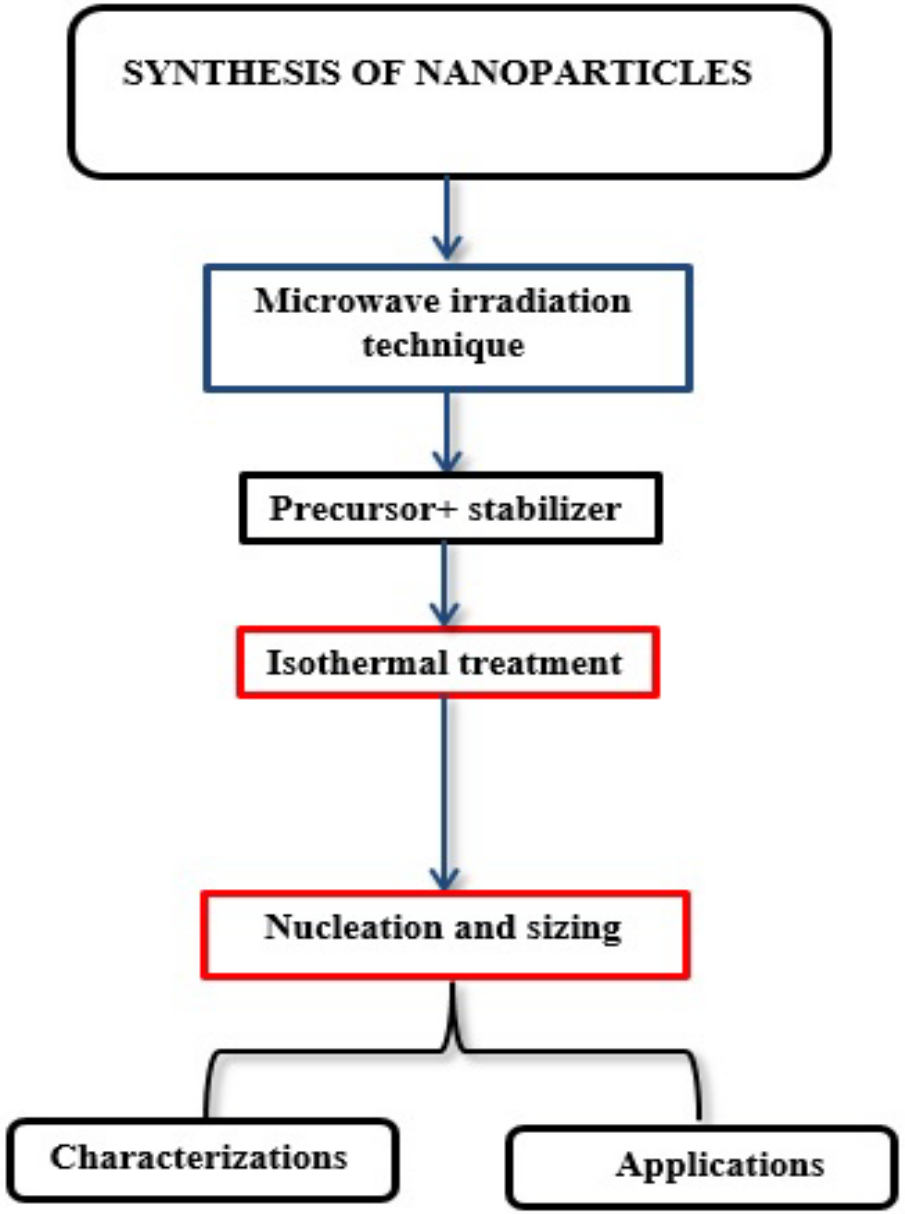
Schematic presentation of Sonolysis synthesis of Iron oxides nanoparticles

- Sonication is the act of applying sound energy to agitate particles in a sample, for various purposes.
- Ultrasonic frequencies of (>20 kHz) are usually used, leading to the process also being known as ultra-sonication.

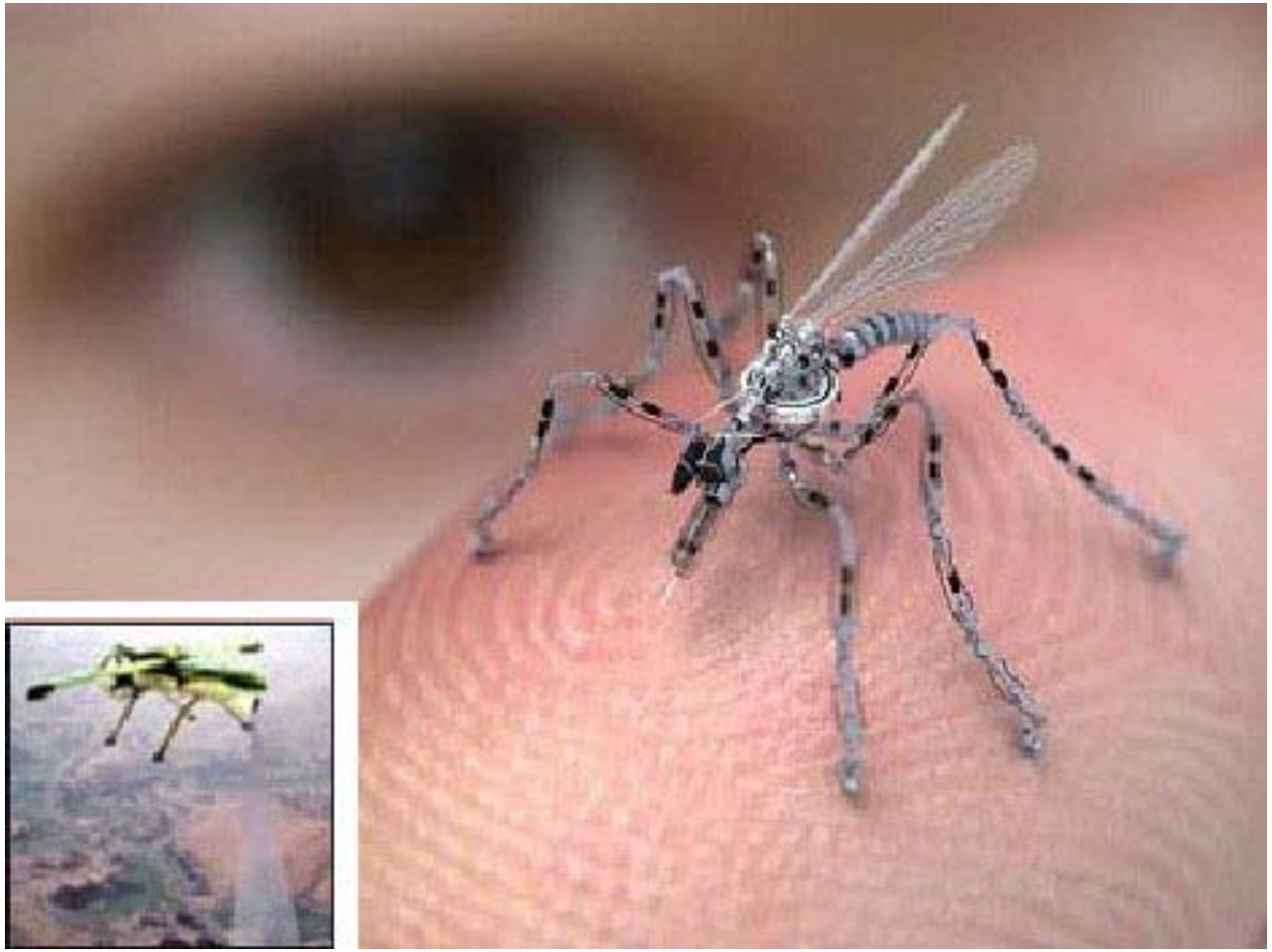
Microwave irradiation



- Uniform heating of precursors, leads to homogenous nucleation and a shorter growing time, may be adopted for large scale production
- The long time processing at high temperature could result in substantial coarsening and grain growth. It could be a unique approach to achieve densification of nano-phase materials.
- Anisotropy arrangement is low resulting in spherical shaped nanoparticles, size ranges from 10-30 nm.



Schematic presentation of Microwave synthesis



The development of Bionic Hornet (flying robot); a war weapon designed to seek out, follow, photograph and even kill selected opponents.