FABRICATION OR SYNTHESIS OF NANOMATERIALS

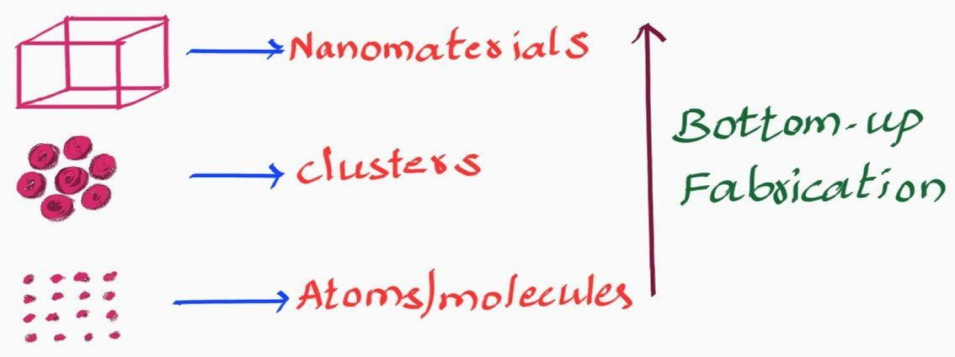
- The fabrication of nanomaterials is the process of creating nanoscale structures and devices using nanomaterials.
- There are various techniques that are capable of creating nanostructures but the general techniques/approaches/methods are:

1.Bottom-up approach/technique/fabrication

2. Top-down approach/technique/fabrication

3.BOTTOM-UP FABRICATION

- Bottom-up fabrication is a nanotechnology approach that uses physical and chemical forces to assemble atoms/molecules into larger, more complex structures on their own.
- It is also known as self-assembly.



- They chemically assemble themselves by recognizing the molecules of their own type.
- This approach (Fabrication) starts by collection and combination of atoms/molecules to build complex structures.
- The methods used to prepare nanomaterials in Bottom-up fabrication are:

(i) Sol-Gel method

(ii) Precipitation method

(iii) Combustion method

(i)SOL-GEL METHOD:

- The sol-gel method is a wet chemical method or chemical solution deposition method.
- This method is used to (generate) produce nanoparticles & nanopowder in the form of oxide material such as TiO_2, ZnO, Al_2O_3 and various rare earth oxides.
- A given material is converted into colloids & dissolved in water/alcohol/acids, then form a solution.
- A colloid suspended in a liquid is called "sol" and a suspension that keeps its shape is called "Gel" (Gelatin).

Principle:

- The basic principle of the sol-gel method is “the formation of a hydroxide through Hydrolysis and Polycondensation reactions of a molecular precursor in a liquid”.

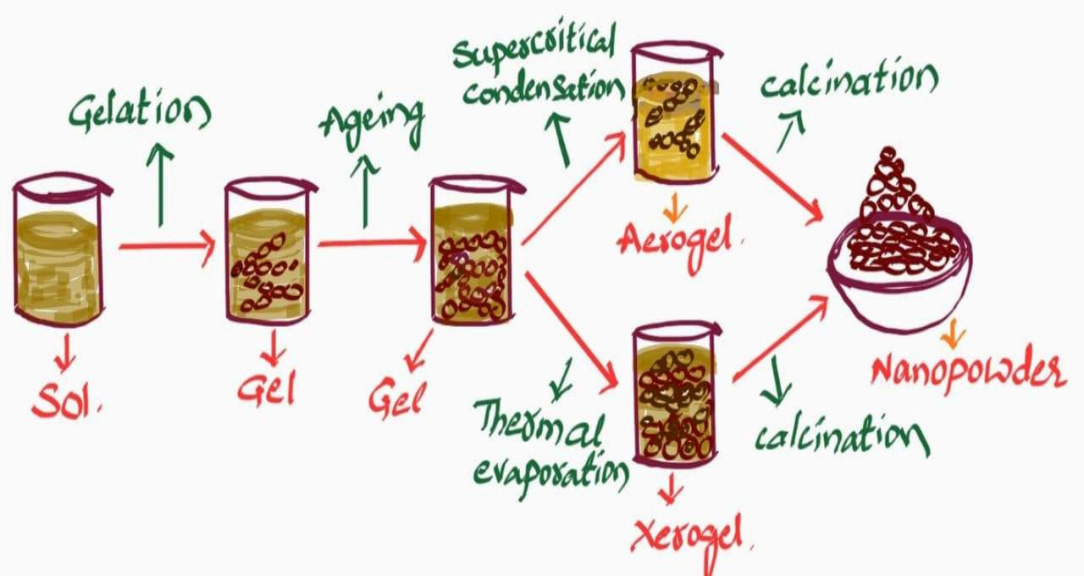
Steps involved in precipitation method:

(i) Formation of Sol. (ii) Gelation (iii) Aging of Gel

(iv) Drying of Gel (v) Calcination

- **The schematic representation of Sol-Gel Method as follows:**

Precursor → Sol→ Gel → Aerogel or Xero Gel → Final product



(**i) Formation Sol :** Take precursors(raw material) like metal alkoxide or metal salts and dissolved in water or alcohol or other solvents.

- It forms a colloidal suspension known as a solution simply "sol".

(ii) Gelation: Gelation can be induced by various methods such as hydrolysis and condensation reactions.

- During **hydrolysis**, the metal alkoxide precursors react with water to form metal hydroxides, while **condensation** reactions lead to the formation of bonds between the nanoparticles, resulting in the formation of a “gel”.

(iii)Aging of Gel: After gelation, the gel is typically allowed to age at a controlled temperature and humidity for a certain period.

- Aging promotes **polycondensation** reactions and allows the gel structure to mature, leading to the formation of solid mass along with contraction of the gel network.

(iv)Drying of Gel: Drying can be carried out through evaporation, freeze-drying, or supercritical fluid extraction to remove the solvents (water and other liquids) from the gel.

- If the rapid drying under **supercritical conditions** results in the formation of aerogel while **thermal evaporation** results in the formation of a xerogel.

(v)Calcination: Calcination is a final step used to transform a dry gel into the desired final material, typically an oxide or ceramic powder

NOTE: Spinning also used to deposit a thin, uniform film of dry gel material onto a substrate.

Advantages:

1.Cheap and low-temperature technique.

2.To prepare mono-sized nanoparticles.

3.Obtain very high purity material.

4.Easy control of the rate of reaction.

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5.Even small quantities of dopants can be introduced.

Disadvantages:

1.Controlling the growth of particles is difficult.

2.Issue with shrinkage and cracking during drying.

3.Issue the potential cost of precursors.

Applications:

1.It can be used for ceramics, thin film manufacturing.

2.These derived materials have applications in optics, electronics, energy, and space.

3.These derived materials also have applications in bio-sensors, medicine, etc.

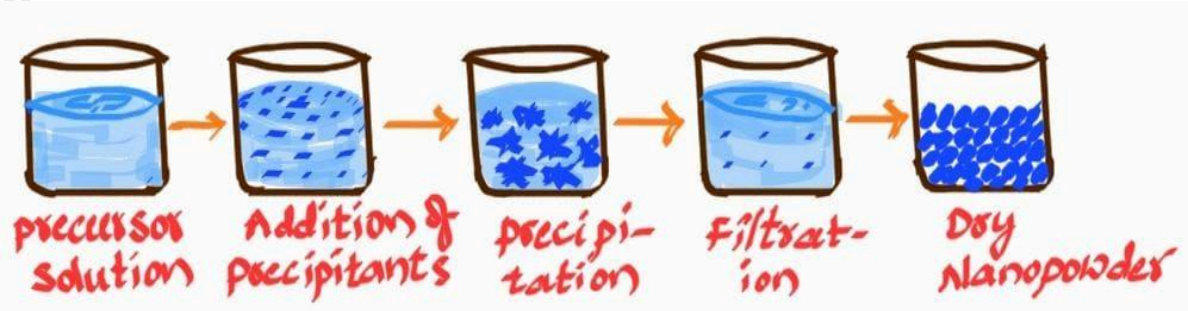
4.It is used to carry out zeolite synthesis.

(ii) PRECIPITATION METHOD:

Precipitation method of fabrication is a technique involving chemical reaction to produce solid particles such as metal oxides or hydroxides or sulphides or other inorganic nanoparticles from a solution. Precipitate is a substance that is deposited in solid form from a solution.

Principle: When two or more chemicals are mixed to react with each other, the product will settle down as precipitate.

Steps involved in precipitation method:



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Preparation of precursor solutions: Prepare precursor solutions which are aqueous and contain metal salts such as chlorides, nitrates, sulphates etc.

Addition of precipitating agent: Slowly add precipitating agents such as strong bases to precursor solutions to initiate the precipitation reaction by causing the formation of insoluble metal hydroxides or other precipitates.

Control reaction parameters/conditions: control the reaction time, pH, conc., temperature to achieve the desired particle size & crystalline structure.

Formation of nanoparticles: As the precipitating agent (precipitant) reacts with the precursor ions, insoluble nanoparticles begin to form in the solution. The nanoparticles nucleate and grow over time as more precursor ions are converted into solid particles (nanoparticles).

Separation & washing: The solid particle suspension is separated from the reaction mixture by centrifugation or filtration. These collected particles are washed several times with solvent to remove residual reactants, impurities or by-products.

Drying & Annealing is an optional process: The washed nanoparticles may be dried at low temperatures and achieve a dry powder form. Annealing (or) calcination may be performed to improve crystallinity.

Advantages:

1. It is a simple, low cost and rapid method for precipitation of nanoparticles.
2. Low reaction temperature & control of particle size.
3. Fine and uniform sized particles are products.
4. Time and energy efficiency.

Disadvantages:

1. Difficult to control the size and shape of the Nanomaterials.

Applications:

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1.synthesizing nanoparticles, nanorods, thin films, and monoliths.

2.This technique allows for the creation of hybrid nanoparticles in various industries.

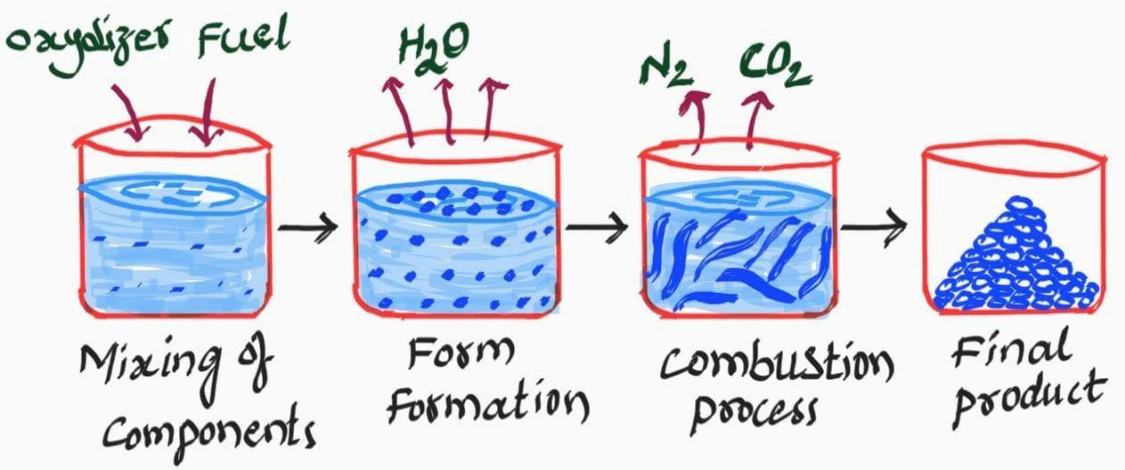
3.It's also a valuable method for synthesizing catalysts, like Ni-Nb-O mixed oxides.

(iii) COMBUSTION METHOD:

- Combustion method of fabrication is a technique involving exothermic reaction between a fuel and oxidizer to generate high temperature and pressures, facilitating the formation of nanoparticles.
- It is a traditional method, discovered by KC Patil.

Principle: This method is based on the principle of utilization of heat energy produced during exothermic (spontaneous redox) reaction between organic fuel & oxidizer such as metal nitrates.

Steps involved in combustion method:



- Preparation of precursors (oxidizer): The process begins with the preparation of precursors, which serves as the source of the desired nanomaterials such as metal nitrates (metal salts).

Mixture Formation: The precursor (oxidizer) materials are mixed with suitable fuel which contain carbon & hydrogen as main components such as glycine ($C2H_5NO_2$), ascorbic acid ($C_6H_8O_6$) and etc.

Ignition and Combustion: The mixture is ignited initiating the combustion reaction. This combustion generates high temperature & pressure, leading to rapid heating and releasing gaseous products such as N_2, CO_2, H_2O. The exothermic nature of this reaction drives the formation of nanoparticles from the mixture.

Quenching: Quenching can be achieved by various methods such as water, air quenching.

Product collection & purification: Collect the nano materials formed in combustion and subjected to purification by washing, filtration & centrifugation.

Advantages:

1.Simple experimental setup & less expensive

2.A molecular level mixing for the raw materials

3.The composition of products can be tuned

Disadvantages:

1. Limitted controlling over particle size, morphology, and structure.

2. Potential issues with powder agglomeration and unwanted products.

Applications:

1. This method offers advantages like simplicity, cost-effectiveness, and rapid synthesis.

2. It's well-suited for creating Metal oxides, Phosphates, Silicates, and Sulfides.

3. It is attractive for various applications like catalysis, energy storage, and biomedical.

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