PREPARATION METHOD OF COLLOIDS AND PURIFICATION OF COLLOIDS

Methods of Preparations and Dispersion Methods

Preparation of Lyophilic sols

Various organic substances, such as gelatin, starch, agar, egg albumin, and glycogen, exhibit ready solubility in water, either at cold temperatures or upon warming, resulting in the direct formation of colloidal solutions. These substances are categorized as lyophilic colloids. For instance, sols of egg albumin or glycogen can be prepared by dissolving 1-2 grams of the finely ground substance in 100 ml of distilled water, followed by allowing it to stand for two hours with constant stirring. After this period, the solutions are filtered. Gelatin, considered a prototypical lyophilic linear colloid, demonstrates unique behavior. When two grams of gelatin are placed in distilled water for several hours, it is observed that, unlike egg albumin and glycogen, gelatin does not dissolve in cold water but undergoes swelling. The swollen gelatin can be dissolved by heating it with water at 80-90°C. If two grams of gelatin are dissolved in 400 ml of distilled water, a clear sol is obtained upon cooling.

Preparation of Lyophobic sols

such sols can be prepared by the two general ways.

1. Through the dispersion method, colloids are formed by breaking down larger particles into colloidal size. This process involves the dispersion of coarse particles to achieve colloidal dimensions.
2. Using the condensation method, colloids are created by encouraging molecular particles to come together and form larger aggregates. In this method, particles of molecular dimensions are initially present and then condensed to achieve colloidal dimensions.

Dispersion Methods

A. Mechanical Dispersion

In this process, the substance is initially finely powdered, and a coarse suspension is created by shaking the powdered substance with the dispersion medium. This suspension is then directed through a colloid mill, comprising two discs moving in opposite directions at an exceptionally high speed (see Fig. I). The particles in the suspension undergo significant shearing force, breaking down to colloidal dimensions. The gap between the two discs controls the size of the colloidal particles obtained. Rubber, ink, paints, and varnishes can be produced using this method.

Another method involves employing a ball mill, as depicted in Fig. (II), to obtain a colloidal solution from a coarse suspension. The high-speed rotation of the mill causes coarse ball-like particles to roll over one another and drop at a specific position within the mill, thereby grinding the initial wetted mixture. This results in the formation of particles with a size of 6 Å. By adding a certain amount of the dispersion medium, it becomes possible to achieve a colloidal solution.

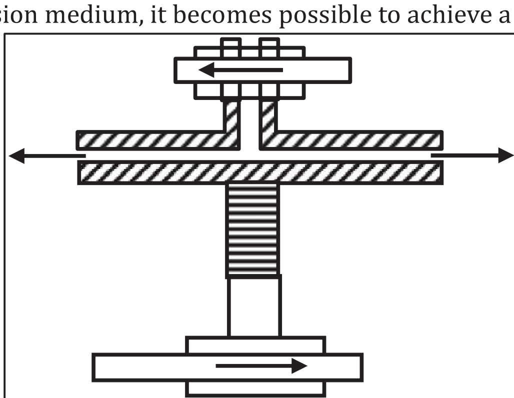


Fig- I

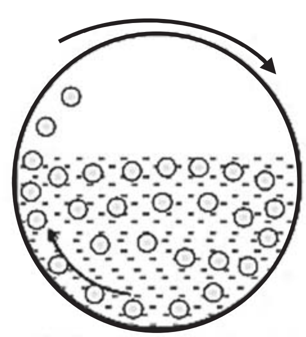


Fig- II A colloid ball mill

B. Electrical Dispersion- Bredie's Arc Method

This method is widely employed for the production of colloidal solutions containing metals. An electric arc is initiated between two metallic rods submerged in the liquid (dispersion medium). Typically, a current of 10 amperes and a voltage ranging from 100 to 300 volts are applied. The liquid is kept cool by surrounding it with a cooling mixture. Minute particles of the metal detach from the rods and disperse in the liquid. Metals like gold, platinum, silver, copper, and others can be obtained in colloidal form using this technique (see Fig. III).

Svedberg introduced a modification to this method, generating sols in non-aqueous media such as pentane and diethyl ether. This modification involves striking an arc with high-frequency alternating current, significantly reducing the decomposition of the liquid (see Fig. IV). Svedberg demonstrated that electrical methods are effective not only for preparing hydrosols of metals like gold, silver, platinum, etc., but also for sols of strongly electropositive metals, such as sodium in benzene.

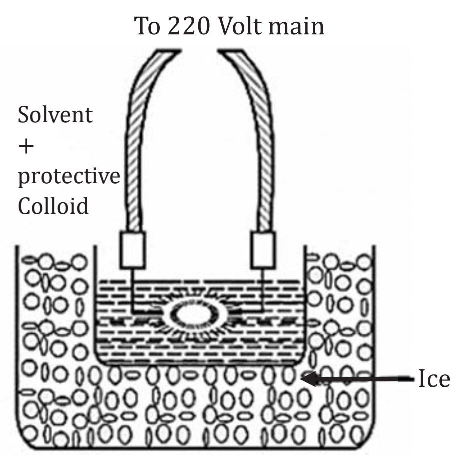


Fig -III

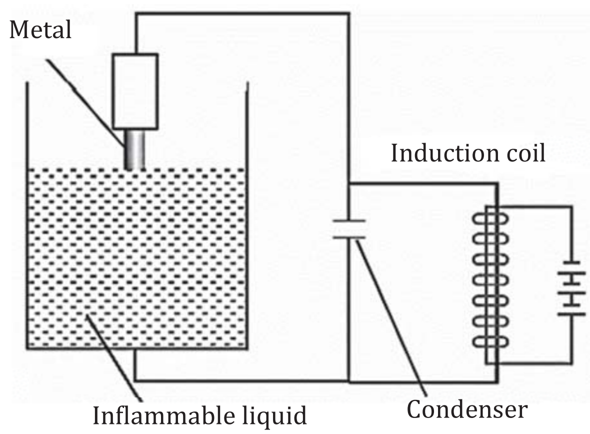


Fig -IV. Svedber's method of preparing metals sols in inflammable liquids

C. Peptization

The procedure of returning a precipitated substance to its colloidal state is referred to as peptization. This is achieved by introducing an electrolyte, termed as a peptizing or dispersing agent. The process entails the adsorption of a suitable ion provided by the electrolyte onto the particles of the precipitate. Peptization can be accomplished through various methods:

i. By electrolyte:

The freshly prepared precipitate of $Fe(OH)_3$ can be transformed into a colloidal state by treating the precipitate with a small quantity of $FeCl_3$ solution. The resulting sol is positively charged, attributed to the preferential adsorption of Fe^{3+} ions (from $FeCl_3$) onto the sol particles of $Fe(OH)_3$ as $[Fe(OH)_3]Fe^{3+}$. It is essential to note that only newly formed precipitates can undergo peptization.

ii. By washing a precipitate:

Peptization can sometimes be induced by repeatedly washing a precipitate. For instance, continuous washing of the $BaSO_4$ precipitate reaches a point where the washings carry some of the substance particles in the form of a colloidal solution.

D. Peptization

Peptization refers to the process of restoring a precipitated substance to its colloidal state, and it is achieved through the addition of an electrolyte, referred to as a peptizing or dispersing agent. This method involves the adsorption of a suitable ion provided by the added electrolyte onto the particles of the precipitate. Peptization can be accomplished through the following methods:

i. By Electrolyte: The freshly prepared precipitate of $Fe(OH)_3$ can undergo transformation into a colloidal state by treating it with a small amount of $FeCl_3$ solution. The resulting sol is positively charged due to the preferential adsorption of Fe^{3+} ions (from $FeCl_3$) onto the sol particles of $Fe(OH)_3$ as $[Fe(OH)_3]Fe^{3+}$. It is essential to note that only freshly prepared precipitates are amenable to peptization.

ii. By Washing a Precipitate: Peptization can sometimes be induced by repetitively washing a precipitate. For example, continuous washing of the $BaSO_4$ precipitate reaches a point where the washings carry some particles of the substance in the form of a colloidal solution.

Condensation Methods

1. Preparation of Lyophilic Sols

The creation of colloidal solutions containing lyophilic colloids such as starch, glue, and gelatin is easily achieved by dissolving these substances in water, either at cold temperatures or upon warming.

2. Preparation of Lyophobic Sols

Lyophobic sols are produced through specialized techniques, which can be categorized into two distinct methods:

(a) Dispersion Methods: Involving the breakdown of coarse aggregates of a substance into colloidal-sized particles.

(b) Condensation Methods: Involving the aggregation of very small particles into colloidal- sized particles.

i. Dispersion method

a. Mechanical dispersion
b. Electro-dispersion
c. Ultrasonic dispersion
d. Peptization

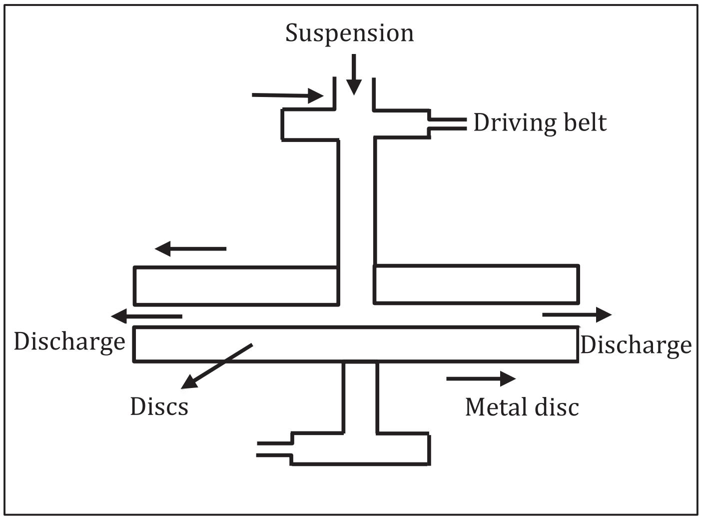
ii. Condensation methods

a. Exchange of solvents
b. Change of physical state
c. Chemical methods
  i. Double decomposition
  ii. Oxidation
  iii. Reduction
  iv. Hydrolysis

i. Dispersion Methods

a. Mechanical Dispersion using Colloidal Mill

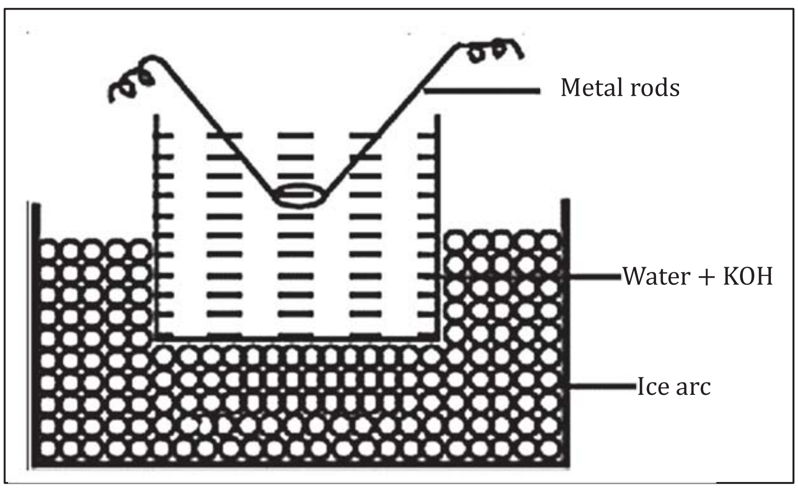
The process involves introducing a mixture of solid material and liquid into a colloidal mill. This mill is equipped with two rotating steel plates that are positioned closely together and move in opposite directions at high speeds. The solid particles undergo a grinding process within the mill, resulting in their reduction to colloidal size. Following this grinding phase, the particles are dispersed within the liquid medium. This technique is particularly utilized for the production of colloidal graphite and printing inks.



Colloidal mill

b. Electro-Dispersion Method: (bredig's Arc Method)

This technique is well-suited for crafting colloidal solutions of metals, such as gold, silver, platinum, and others. The process involves initiating an arc between metal electrodes submerged beneath the water's surface, with the water containing a stabilizing agent like a trace of alkali. To control the temperature, the water in the container is cooled by immersing it in a cold bath. The high heat generated by the arc leads to the vaporization of some of the metal, and this vapor then condenses when brought into contact with the cold water.



Bredin's arc method

c. Ultra-Sonic Dispersion

Ultra-sonic waves, characterized by their high frequency, play a crucial role in this method. Specifically termed ultra-sonic waves, they are directed through a solution that contains larger particles. As a result of this ultrasonic treatment, the larger particles break down, leading to the formation of a colloidal solution.

d. Peptization

Peptization involves the dispersion of a precipitated material into a colloidal solution through the influence of an electrolyte within the solution, known as a peptizing agent. In this process, certain examples highlight its application: for instance, silver chloride can be transformed into a sol by the addition of hydrochloric acid, and ferric hydroxide can be converted into a sol by introducing ferric chloride.

ii. Condensation Methods

a. By Exchange of Solvent

When a solution of sulfur or phosphorus in alcohol is introduced into water, a colloidal solution of sulfur or phosphorus is produced due to their low solubility in water.

b. By Change of Physical State

Colloidal solutions of specific elements, like mercury and sulfur, are acquired by passing their vapor through cold water that contains a stabilizer.

c. Chemical Methods

Chemical methods entail chemical reactions in a medium where the dispersed phase is sparingly soluble. Various methods fall under this category.

i. Double Decomposition

To create an arsenic-supplied sol, a slow stream of hydrogen gas is passed through a cold solution of arsenious oxide until the yellow color of the sol reaches maximum intensity. The reaction is represented as follows:

$$as_2o_3 + 3\ h_2s \text{ --- --- -- > } as_2s_{3(yellow)} + 3\ h_2o$$

Any excess hydrogen is removed by passing a stream of hydrogen.

ii. Oxidation

A colloidal solution of sulfur is obtained by introducing hydrogen sulfide (H_2SH_2S) into a solution of sulfur dioxide:

$$2h_2s + so_2 \text{ ---- -- - > } 2h_2o + 3\ s$$

iii. Reduction

Silver sols and gold sols can be obtained by treating a dilute solution of silver nitrate or gold chloride with organic reducing agents like tannic acid or formaldehyde.

The reactions are as follows:

$$agno_3 + \text{tannic acid} \text{ --- -- > } \text{ag sol}$$

$$auno_3 + \text{tannic acid} \text{ --- -- > } \text{au sol}$$

iv. Hydrolysis

Colloidal solutions of the hydroxides of Fe, Cr, Al, etc., can be prepared by the hydrolysis of their salts. For example, a colloidal solution of ferric hydroxide is obtained by boiling a dilute solution of ferric chloride:

$$fecl_3 + 3h_2o \text{ -- --- - -> } fe(oh)_{3(red\ sol)} + 3hcl$$

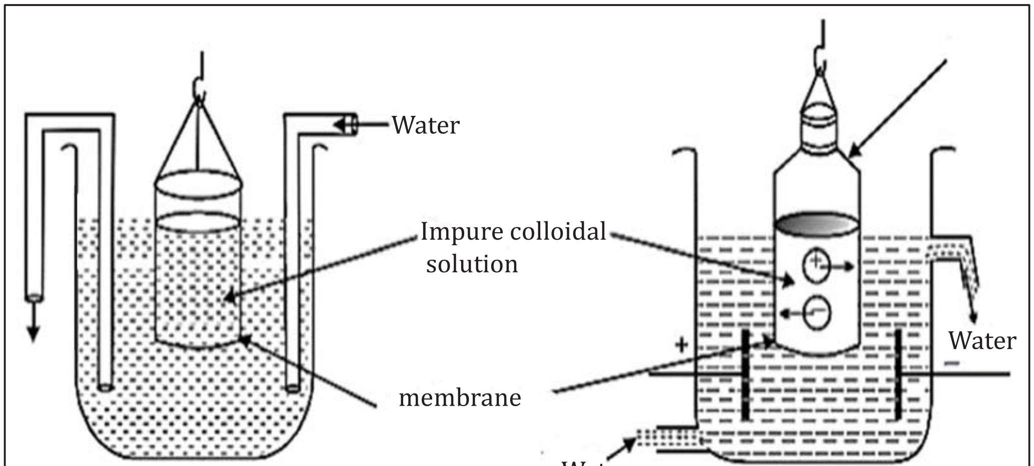
Purification of Colloidal Solution

Purification of sols

A sol often retains elevated levels of electrolytes and certain other soluble impurities due to the chosen preparation method, especially in chemical condensation techniques.

1. **Dialysis**

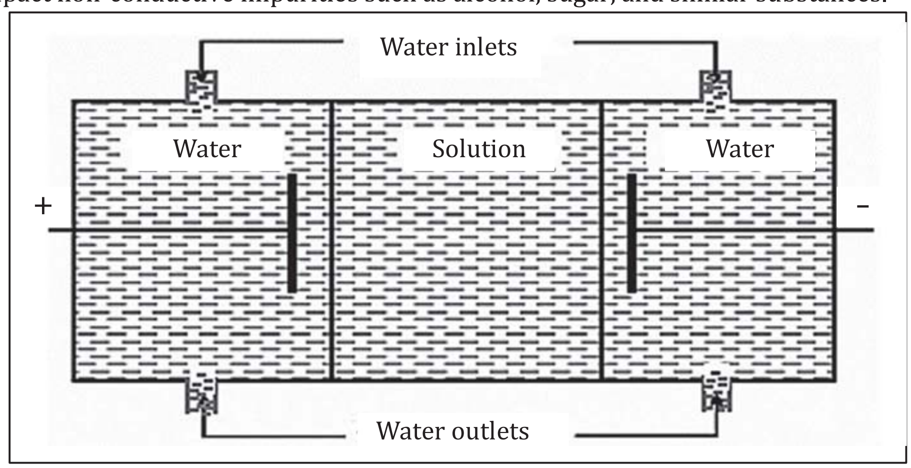
This technique relies on the principle that colloidal particles are held back by animal membranes or parchment paper, whereas electrolytes can pass through them. The sol is placed in a parchment or cellophane bag, which is then immersed in flowing water in a trough. Over time, the soluble impurities gradually diffuse out, leaving a purified sol. Dialysis is a gradual process, often requiring several hours and sometimes even days for complete purification.



(a) Dialysis (b) Electrodialysis

2. **Electrodialysis**

Dialysis can be expedited by introducing an electric field, particularly when the substance in true solution is an electrolyte. This variant of the process is termed electrodialysis. Through electrodialysis, it becomes feasible to rapidly obtain a pure colloid, although it's noteworthy that the electric current doesn't impact non-conductive impurities such as alcohol, sugar, and similar substances.



3. **Ultra-filtration**

This technique serves not only for the purification of the sol but also for its concentration. The ordinary filter paper's pores, with a size of 10-30 μm, are sufficiently large for colloidal particles (20-3 μm) to pass through. However, by reducing the pore size, colloidal particles can be retained on the filter paper, and this process is termed ultra-filtration.

4. **Electro Decantation**

This method, effective for both sol purification and concentration, involves electrodialysis without stirring the sol. Consequently, the lower layer becomes more concentrated, while the top layer becomes dilute. Introduced by Pauli, this process is known as electro decantation.

5. **Ultracentrifuging**

Colloidal particles exhibit continuous zigzag motion, known as Brownian movement, as they share the motion of the molecules in the dispersion medium. This motion prevents sol particles from settling. In ultracentrifugation, the sol is placed in a high-speed centrifuge rotating at about 15,000 revolutions per minute. This rapid rotation causes colloidal particles to settle quickly, and the resulting slime can be suspended in water to obtain a sol