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## 3. Preparation and Purification of Colloids

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### Introduction:

Colloids are an important part of our everyday life and play an important role in the fields of agriculture, medicine, biology, industries, etc. Proteins, starch, gelatin, jellies, milk, curd, cheese, paints, varnishes, alloys, smoke, clouds, fog, volcanic dusts, etc. are familiar examples of different types of colloids. Hence a knowledge and understanding of colloidal chemistry is essential.

A *colloidal solution* or *colloidal dispersion* is a mixture of two substances in which one substance is present in form of finely divided particles (with diameter lying between 10–2000 Å) that are dispersed in the other substance.

Thus, a colloidal solution is a two phase system. The substance present as minute particles forms the discontinuous phase called the *dispersed phase* while the substance in which the particles are dispersed forms the continuous phase, referred to as the *dispersion medium*. For example- in a colloidal solution of gold in water, the gold particles constitute the dispersed phase and water forms the dispersion medium. Both the dispersed phase and dispersion medium can either be a solid or a liquid or a gas. So there are eight different types of colloidal systems.

Out of the different types of colloidal systems, the three main types are –

- Sols* in which a solid is dispersed in a liquid. For e.g.- proteins, paints, gold sol, arsenious sulphide sol, etc.
- Emulsions* in which both the dispersed phase and the dispersion medium are liquids. For e.g.- milk, cod-liver oil, butter, etc.
- Gels* in which a liquid is dispersed in a solid. For e.g.- gelatin, jellies, silica gel, etc.

In this chapter, methods for the preparation of sols, emulsions and gels will be discussed along with the techniques used for the purification of sols.

### 3.1 Preparation of Colloids:

A colloidal solution is intermediate between a true solution and a suspension. This is because the diameter of the dispersed particles in a colloidal system is more than that of the solute particles in a true solution (with diameter between 1-10 Å) but smaller than that of a suspension (with diameter of more than 2000 Å). Hence the main consideration in preparation of colloidal solutions is that the dispersed particles should be in the size range of 10-2000 Å.

### 3.1.1 Sols:

A colloidal system in which the dispersed phase is a solid and the dispersion medium is a liquid is called *sol*. When the dispersion medium is water, the sol is known as *hydrosol* and if it is alcohol or benzene or any other organic solvent, the sol is named *alcosol* or *benzosol* or *organosol* respectively.

On the basis of the affinity of the dispersed phase for the dispersion medium, sols have been divided into two categories:

- a. **Lyophilic sols** (*liquid loving*) in which the dispersed phase exhibits strong affinity for the dispersion medium. For e.g.- proteins, gum in water. If water is the dispersion medium, lyophilic sols can also be termed as hydrophilic sols.
- b. **Lyophobic sols** (*liquid hating*) in which the dispersed phase has no affinity for the dispersion medium. For e.g.- gold sol, ferric hydroxide sol, etc. When water is the dispersion medium, the lyophobic sols are also called hydrophobic sols.

Due to the difference in the affinity between the two phases in the lyophilic and lyophobic sols, different methods are employed for their preparation.

### 3.2 Preparation of Lyophilic Sols:

In lyophilic sols (also called *intrinsic colloids*) the dispersed phase shows strong affinity for the dispersion medium. Hence they can be easily prepared by simply mixing the solid (to be dispersed) with the suitable liquid acting as the dispersion medium.

In some instances slight warming may also be required to obtain the sol. These sols are stable and, therefore, addition of stabilizers is not required during their preparation. They are reversible in nature, since on evaporation of the dispersion medium, the residue left can again be converted into colloidal state by addition of the liquid.

Some examples of lyophilic sols are: starch, gelatin, acacia in water, rubber in benzene, etc.

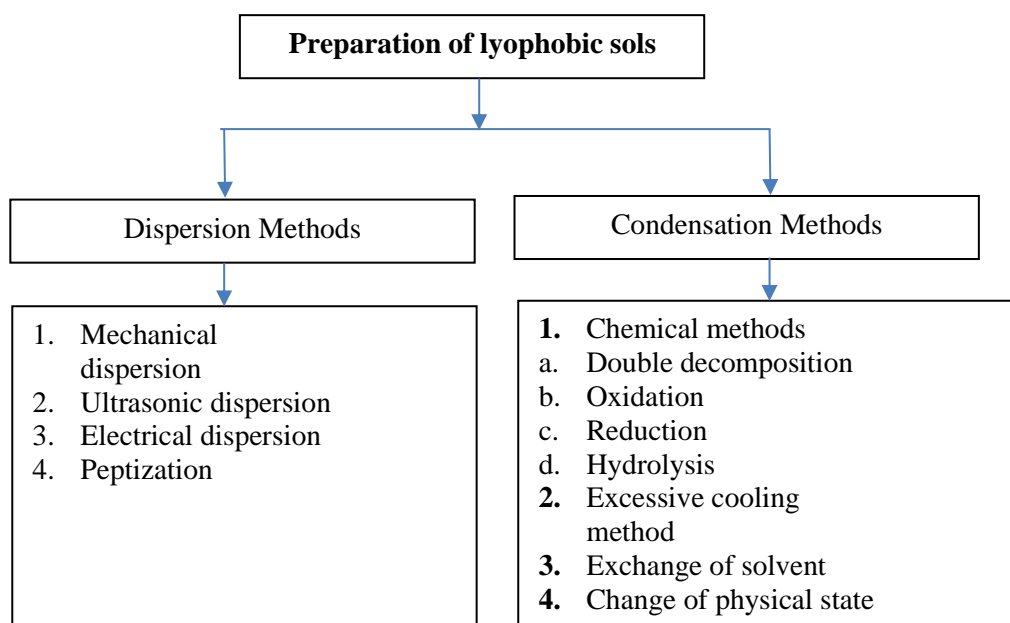
### Preparation of Lyophobic Sols:

Lyophobic sols (also called *extrinsic colloids*) cannot be prepared by simply mixing the solid with the liquid medium since there is hardly any affinity between the dispersed phase and the dispersion medium.

They are comparatively unstable and hence require stabilizing agents at the time of preparation. They are irreversible in nature which means that once the dispersion medium is evaporated, the residue left cannot be converted again into sol by addition of the liquid.

Therefore, special techniques are employed for the preparation of lyophobic sols. The methods for their preparation have been divided into two categories:

1. **Dispersion methods:** They involve breaking down of bigger sized particles of a substance into smaller particles of colloidal dimensions. The colloidal particles obtained are stabilized by the addition of a stabilizing agent.
2. **Condensation / Aggregation methods:** In these methods, particles of colloidal size are built by the aggregation of a large number of atoms or molecules.



### 3.2.1 Dispersion Methods:

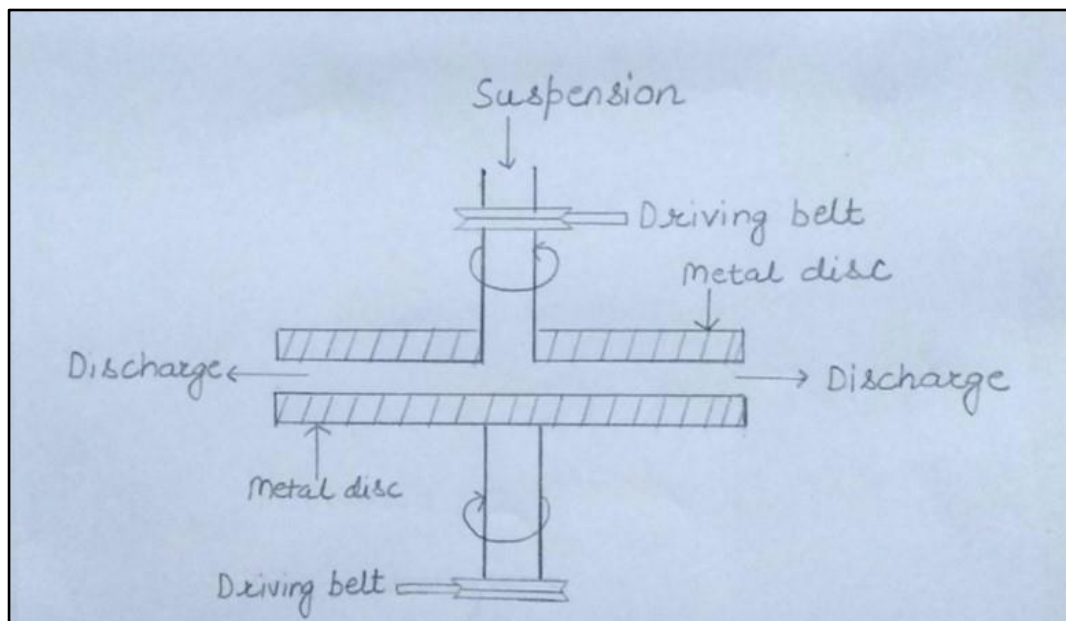
The different types of dispersion methods used for the preparation of lyophobic sols are:

#### A. Mechanical dispersion:

In this technique the substance (to be dispersed) is first ground by usual methods to obtain smaller sized particles. It is then mixed with the dispersion medium to get a suspension. This suspension, together with a stabilizing agent, is then passed through a *colloidal mill* (Figure 3.1).

The colloidal mill, also known as *disc mill*, consists of two heavy metal discs placed one above the other and separated by a very small distance. These discs are capable of rotating at a very high speed (about 7000 revolutions per minute) in opposite directions. The powerful shearing force breaks the suspended particles into colloidal sized particles and thus a sol is obtained.

**Some examples:** Colloidal solutions of printing inks, paints, varnishes, etc. are prepared by this method.



**Figure 3.1: Colloidal Mill**

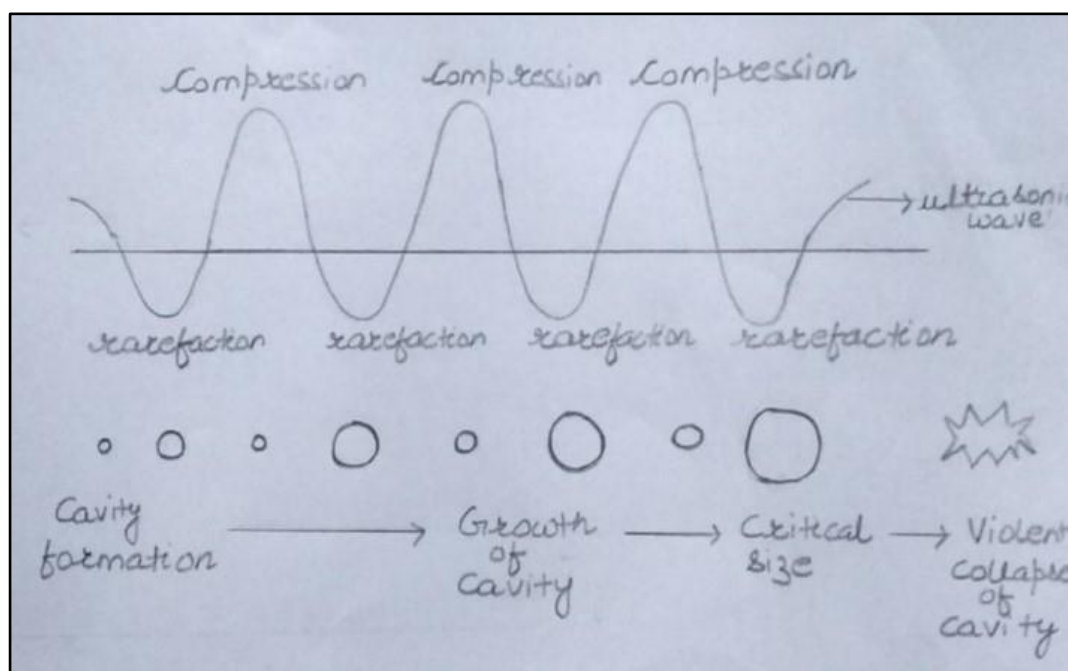
### **B. Ultrasonic Dispersion:**

This method involves the use of high intensity ultrasonic waves and it has been effectively used for the preparation of some sols. Ultrasonic waves refer to ultrasound waves with a frequency of 20 KHz and above (more than the upper limit of human audible range). The ultrasonic waves are usually generated by 'Piezoelectric oscillator' method in which a piezoelectric crystal (such as a quartz crystal) converts electrical energy (electric current) into mechanical energy (sound waves). These waves can also be produced by 'Magnetostriction oscillator' method which involves the use of a ferromagnetic rod of nickel or iron which is magnetized. Due to magnetization there is a small change in the dimensions of the rod which then undergoes rapid vibrations, thereby generating the ultrasonic waves.

Ultrasonic vibrations can transform a coarse suspension into colloidal dimensions. When ultrasonic waves enter the liquid (dispersion medium), they propagate through the liquid by creating alternate compression (contraction) and rarefaction (expansion) cycles (Figure 3.2). This produces a negative pressure in the medium which breaks the liquid into voids, which then form small sized bubbles or cavities. This is called cavitation. The cavities expand and contract with the compression and rarefaction cycles. They continue to grow in size and after attaining a critical size they implode with the surrounding liquid rushing into them. When the cavity collapses violently near a solid surface, high speed jets of liquid with velocity as high as 400 km / hour are formed.

The potential energy of the cavity gets converted into the kinetic energy of the liquid jet. These jets strike the solid surface with great impact. A tremendous shearing force is generated which disrupts the solid and converts it into a colloidal dispersion.

Examples: Claus prepared mercury sol by this method. Also sulphides and metal oxides can be dispersed into colloidal state by this technique.



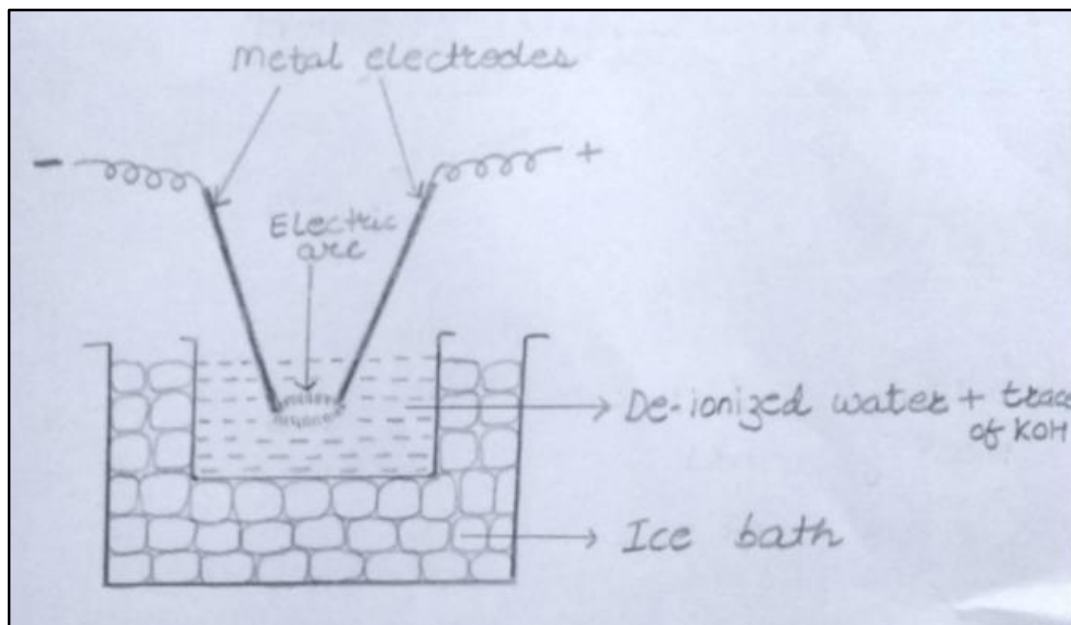
**Figure 3.2: Principle of Ultrasonic Cavitation**

**B. Electrical Dispersion (Bredig's arc method):**

G. Bredig introduced this method in 1898. This technique is used to prepare sols of metals in water (also called hydrosols). Two electrodes of the metal, whose sol is to be prepared, are dipped in *de-ionized* water containing traces of an alkali (KOH). The water is kept cold by immersing the container in an ice bath (Figure 3. 3). The electrodes are connected to a high voltage source. An electrical arc is struck between the two electrodes. The intense heat produced turns some part of the metal into vapours which are immediately condensed by the surrounding ice cold water to give aggregates of colloidal size. Traces of electrolyte added to water (KOH) help in stabilization of the sol.

Examples: Hydrosols of gold, silver, platinum, copper, etc. are prepared by this method.

This technique of electrical dispersion has also been modified to prepare organosols of metals and non-metals. In the modified method, electrodes usually made of iron or aluminium (which do not disintegrate) are employed. The material to be dispersed is suspended in form of granules in the dispersion medium (an organic medium) and an arc is struck between the electrodes. It is noteworthy that this electrical method involves both dispersion as well as condensation processes. The atoms of the metal in vapour state condense to form aggregates of colloidal size but since the process is initiated using bulk metal, hence it is regarded as a dispersion method.



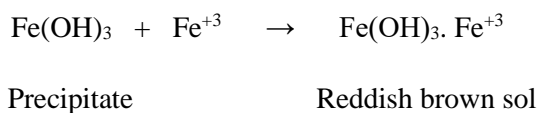
**Figure 3.3: Bredig's arc method**

**D. Peptization:** Certain freshly prepared precipitates such as silver chloride, ferric hydroxide, etc. can be converted into their colloidal solutions in water by the addition of a small amount of a suitable electrolyte. The electrolyte which is added should have an ion common with the precipitate which is to be dispersed. The precipitate preferentially adsorbs one of the ions of the electrolyte which is chemically closely related to it. As a result the particles of the precipitate acquire a positive or negative charge depending on the charge of the ion that has been adsorbed. Due to the presence of similar type of charges, the particles of the precipitate repel each other and are pushed apart. They separate out as colloidal sized particles and in this way a stable sol is formed.

*The process of disintegration of a freshly prepared precipitate into particles of colloidal size by the addition of a small amount of a suitable electrolyte is called peptization. The electrolyte used for this purpose is called peptizing agent. Peptization is the reverse process of coagulation.*

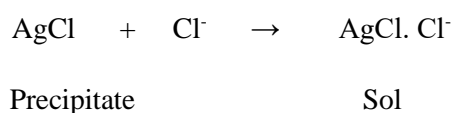
Some examples of sols prepared by peptization are:

Reddish brown colloidal solution of ferric hydroxide ( $\text{Fe}(\text{OH})_3$ ) is obtained on the addition of a small amount of ferric chloride ( $\text{FeCl}_3$ ) to a freshly prepared precipitate of ferric hydroxide. Ferric chloride acts as the peptizing agent (with  $\text{Fe}^{+3}$  ions being in common with the precipitate) and the peptization action is due to the preferential adsorption of ferric ions.



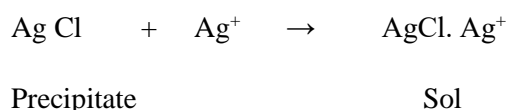
• **Formation of  $\text{Fe}(\text{OH})_3$  sol by peptization:**

Precipitate of silver chloride ( $\text{Ag}^+\text{Cl}^-$ ) yields a sol on the addition of a small amount of hydrochloric acid which acts as the peptizing agent ( $\text{Cl}^-$  ion being in common with the precipitate). Colloidal solution is obtained due to the preferential adsorption  $\text{Cl}^-$  ions.

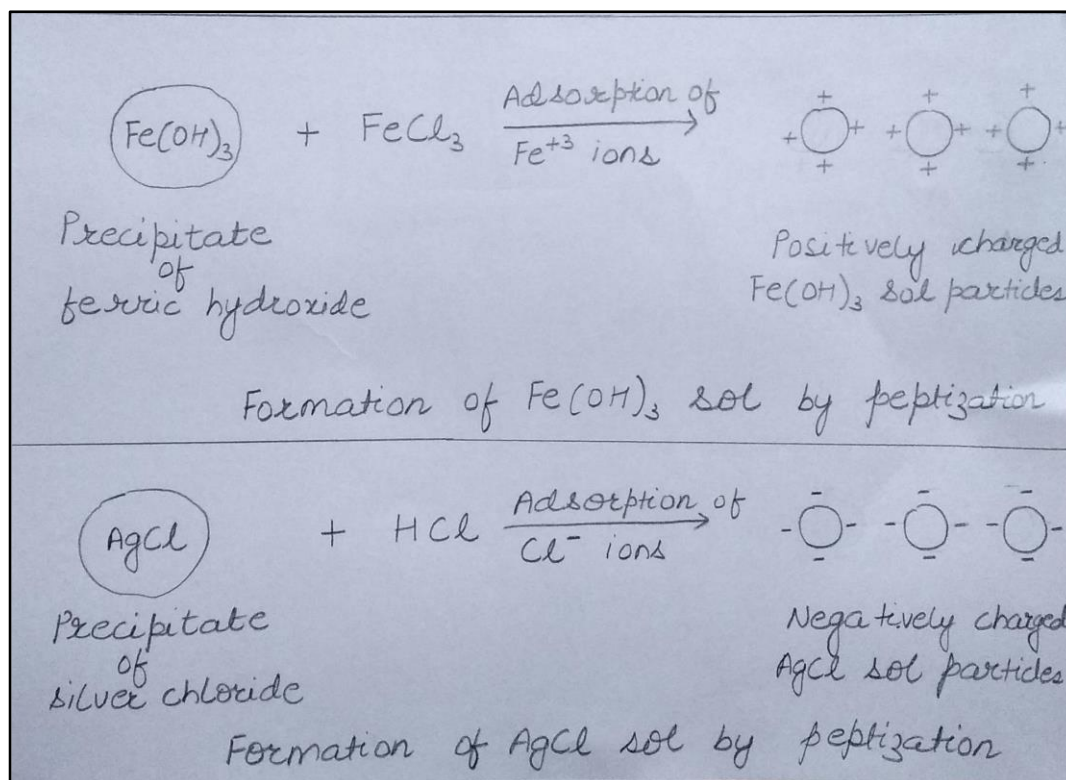


• **Formation of  $\text{AgCl}$  sol by peptization:**

Silver chloride precipitate can also undergo peptization on addition of a small amount of a dilute solution of silver nitrate. Silver ions, being in common, are preferentially adsorbed by the precipitate.



Cadmium sulphide ( $\text{CdS}$ ) precipitate can be converted into a sol by the addition of hydrogen sulphide (sulphide ions being in common are adsorbed).





Sol of aluminium hydroxide ( $\text{Al}(\text{OH})_3$ ) can be obtained by the addition of a small amount of dilute hydrochloric acid to a freshly prepared precipitate of aluminium hydroxide. The action of hydrochloric acid on  $\text{Al}(\text{OH})_3$  yields  $\text{Al}^{+3}$  ions that are preferentially adsorbed. Sometimes repeated washing of the precipitate also causes peptization. For example- when freshly prepared precipitates such as that of  $\text{Al}(\text{OH})_3$ ,  $\text{BaSO}_4$  or  $\text{CuS}$  are repeatedly washed with distilled water, their colloidal solutions in water are obtained.

### **3.2.2 Condensation / Aggregation Methods:**

The principle of condensation methods is that the materials from which the lyophobic sol is to be prepared are present initially in true solution as ions or molecules and they are then converted into particles of colloidal size, by aggregation, using different chemical reactions and methods. These reactions are strictly carried out under controlled conditions of concentration, temperature, order of mixing, etc. that allow the formation of colloidal solutions only but prevent the further growth of the colloidal particles and their subsequent precipitation.

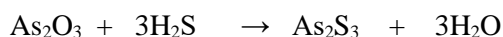
The various types of condensation methods that are commonly used are as follows:

#### **A. Chemical Methods:**

Different types of chemical reactions have been used for the preparation of lyophobic sols.

**a. Double Decomposition:** Various colloidal solutions have been prepared through this reaction. Some examples are as follows:

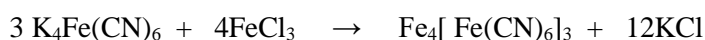
a. A yellow coloured stable sol of arsenious sulphide ( $\text{As}_2\text{S}_3$ ) is prepared by slowly passing hydrogen sulphide gas through a cold solution of arsenious oxide in water.



Yellow sol

The  $\text{H}_2\text{S}$  gas is continuously passed till the yellow colour of sol attains maximum intensity. However, excess of hydrogen sulphide (an electrolyte) is removed either by boiling or by passing in a stream of purified hydrogen or nitrogen or carbon dioxide.

b. On mixing a very dilute solution of ferric chloride and potassium ferrocyanide, a prussian blue sol of ferric ferrocyanide is obtained.

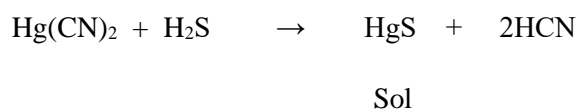


Prussian blue sol

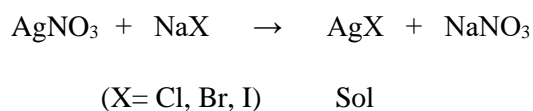
The ionic impurities formed in this reaction are removed by the process of dialysis (discussed later in the chapter).



c. Mercuric sulphide sol is obtained by passing H<sub>2</sub>S gas through a solution of mercuric cyanide.

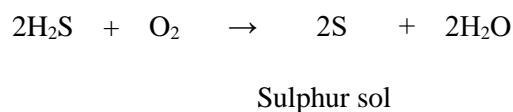


d. Silver halide sols can be obtained by mixing dilute solutions of an alkali halide and a silver salt.

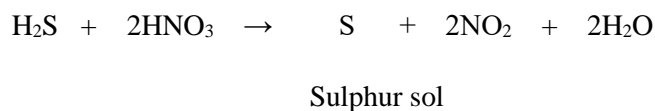
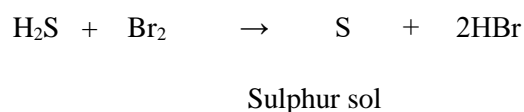
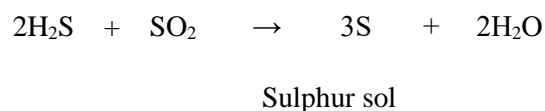


**b. Oxidation:** Certain Sols have been prepared *via* oxidation reactions. For instance:

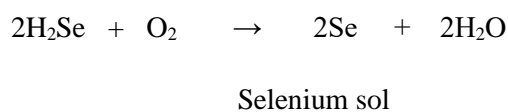
a. On exposing an aqueous solution of hydrogen sulphide to air, sol of sulphur is obtained as a result of an oxidation reaction.



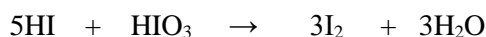
Sulphur sol can also be obtained by passing H<sub>2</sub>S gas through an oxidizing agent such as a solution of sulphur dioxide or bromine water or nitric acid, etc.



b. Selenium sol can be prepared in the same manner by the oxidation of hydrogen selenide solution.



c. Iodine sol is obtained by the reaction of iodic acid with hydroiodic acid.



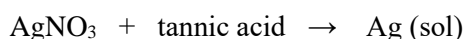
Iodine sol

- c. **Reduction:** Metal sols such as silver, gold, platinum, palladium, iridium, copper, lead, etc. can be obtained by the reduction of the solutions of their soluble salts or oxides. For this purpose, reducing agents that are employed are non- electrolytes.

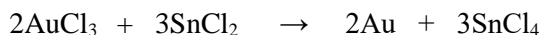
Some examples of the commonly used reducing agents are: formaldehyde, tannic acid, phenyl hydrazine, hydroxylamine, stannous chloride, hydrogen, phosphorus, carbon monoxide.

Some examples of sols prepared by this method are:

- a. Silver sol is obtained by the reduction of silver nitrate with tannic acid.

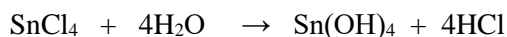


- b. Gold sol has been prepared by the reduction of gold salts, viz, gold (III) chloride with stannous chloride.



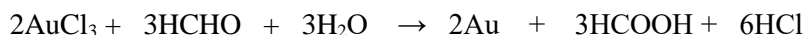
Gold sol

The colloidal gold particles obtained by this reaction are adsorbed on to stannic hydroxide (hydrated stannic oxide) formed by the hydrolysis of  $\text{SnCl}_4$ .



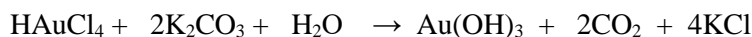
The gold sol, thus obtained, has a purple colour and it is called *purple of cassius* (after the name of Andreas Cassius of Leyden who discovered it in 1685). The intensity of the sol colour correlates with the concentration of gold present. Purple of cassius is quiet expensive and is used to make ruby glass, fine potteries, miniature paintings, etc.

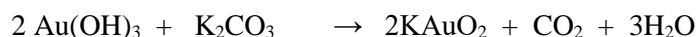
Gold sol can also be prepared by the reduction of a gold salt with formaldehyde.



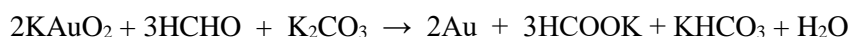
Gold sol

In another method, gold sol has been readily obtained by the reduction of chloroauric acid solution (having traces of potassium carbonate) with formaldehyde. In this reaction, first chloroauric acid reacts with potassium carbonate to yield potassium aurate.



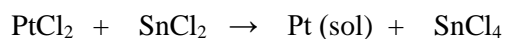


The above solution is heated and a dilute formaldehyde solution is added to it. Reduction occurs and gold sol is obtained.



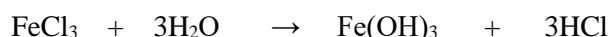
Sol

c. Platinum sol has been prepared by the reduction of its salt solution with a suitable reducing agent.



**d. Hydrolysis:** Sols of hydroxides of weakly electropositive metals such as aluminium, iron, chromium, tin, thorium, zirconium, etc. have been prepared by hydrolysis of aqueous solutions of their salts. The solutions of these metal salts undergo appreciable hydrolysis since the bases corresponding to the metals are weak. Hydrolysis is a type of a double decomposition reaction with water acting as one of the reactants.

**For example:** Red sol of ferric hydroxide is obtained on adding a small amount of ferric chloride solution drop by drop to hot water.



Red sol

In this reaction ferric chloride also acts as a stabilizer. The ferric ions ( $\text{Fe}^{+3}$ ) furnished by  $\text{FeCl}_3$  solution, are adsorbed on the surface of the particles of  $\text{Fe(OH)}_3$  which, resultantly acquire a positive charge.

Presence of similar charge on the particles develops repulsion between them and hence prevents their coagulation. This confers stability to the sol. Purification of sol and removal of HCl formed in the reaction is done by dialysis.

### **B. Excessive cooling method:**

This technique has been used to obtain a colloidal solution of ice in organic solvents such as chloroform or ether. The process involves freezing a solution of water in the organic solvent. Due to freezing the molecules of water, that can no longer be held in solution, separately combine to form bigger particles of colloidal dimensions.

**C. By exchange of solvents:** Colloidal solutions of many substances can be prepared by exchange of solvents. The process involves taking the solution of the substance (to be dispersed) in one solvent and then pouring it into another solvent in which the substance is comparatively less soluble (or sparingly soluble). For instance:

- a. When an alcoholic solution of sulphur is poured in excess of water, a colloidal solution of sulphur in water is obtained. Sulphur is present in molecular form in alcohol but due to its insolubility in water, the molecules precipitate out as colloidal particles in water.
- b. Phenolphthalein, used as an indicator in acid- base titrations, is used in form of its alcoholic solution since it is soluble in alcohol but not in water. Therefore, when water is added to a solution of phenolphthalein, a milky liquid is formed which is actually a colloidal solution of phenolphthalein in water.

**D. By change of physical state:** Colloidal solutions of some elements such as mercury and sulphur have been obtained by this technique. In this method, the vapours of the element are passed through cold water containing a suitable stabilizer (for e.g.- an ammonium salt or a citrate).

### **3.3 Purification of Sols:**

The sols prepared by the methods discussed above usually contain impurities, particularly that of electrolytes. Presence of small amount of electrolytes stabilizes the sol but their excess concentration renders the sol unstable. Hence elimination of these impurities by suitable means is necessary. Some commonly used methods for purification of sols are: *Dialysis, Electrodialysis, Ultra-filtration and Ultracentrifugation.*

#### **A. Dialysis:**

*The process of separating colloidal particles from dissolved ions or molecules of smaller dimensions (crystalloids) by diffusion through a semi- permeable membrane is called dialysis and the membrane used for this purpose is called dialyser*

The technique of dialysis was first employed by British Chemist, Thomas Graham in 1861. The principle of this technique is that while ions or molecules (crystalloids) can easily diffuse through parchment and other fine membranes, the colloidal particles (being bigger in size) cannot do so readily. Hence crystalloids, present as impurities in sol, easily pass through the dialyser but the colloidal particles are retained (due to their very slow rate of diffusion).

Parchment paper is commonly used in dialysis. In addition to it, other membranes that can be used are of cellophane, cellulose nitrate and cellulose acetate. The membranes can be employed in form of a bag or an elongated tube.

The simplest dialyser consists of a bag made of parchment or cellophane in which the colloidal solution to be purified is taken (Figure 3.4). The bag is suspended in a wider vessel containing distilled water. The crystalloids present as impurities in the colloidal solution diffuse out into the surrounding water in the vessel while the colloidal particles remain in the bag. Thus the sol is purified. Accumulation of crystalloids in water may lead to back diffusion, hence the distilled water in the wider vessel is periodically renewed. But it is more preferred to maintain a continuous flow of water in the vessel.

This is achieved by allowing water to enter the vessel from an inlet near the bottom and allowing it to leave from an outlet near the top of the vessel. This accelerates the process of diffusion as the concentration of crystalloids in the surrounding water remains practically zero. This concentration gradient, in turn, favours the fast diffusion of ions or molecules into the surrounding water.

For example: Ferric hydroxide sol prepared by hydrolysis (by addition of ferric chloride solution to hot water) is purified by this method. For the stability of this sol, it is necessary to remove HCl (an electrolyte) formed in the reaction.

Hence, the impure sol of ferric hydroxide is subjected to dialysis. After sometime the entire HCl diffuses into the surrounding water and pure reddish sol of  $\text{Fe}(\text{OH})_3$  is left in the bag.

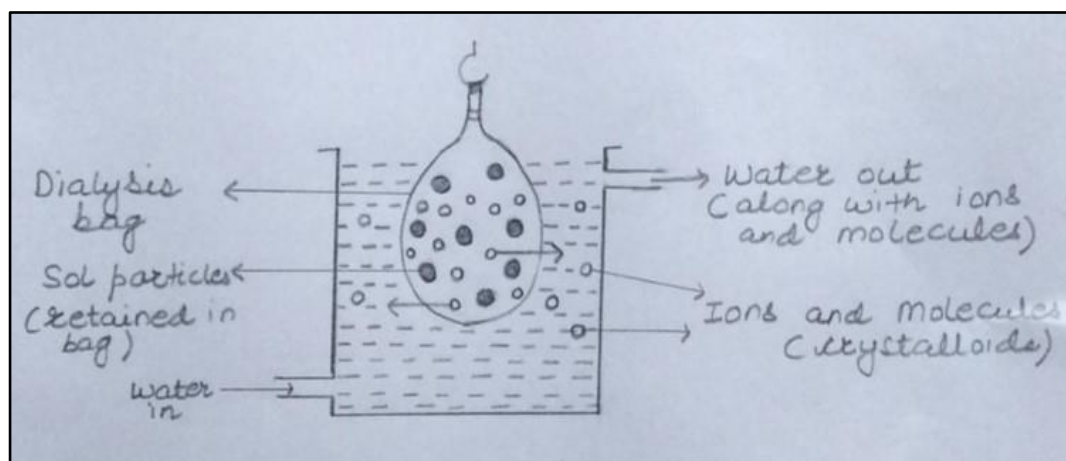


Figure 3.4: Dialysis

### B. Electrodialysis:

The process of dialysis is very slow. However, it can be accelerated by the application of an electric field. This process is referred to as electrodialysis. In this method, two metal screens supporting the dialysing membranes are placed in a container (Figure 3.5). The colloidal solution, to be purified, is placed between the two membranes.

On the other side of each dialysing membrane is a compartment filled with pure water. Required potential is applied between the two metal screens. Under the influence of the electric field, the ions of the electrolyte (present as impurities) migrate towards the oppositely charged electrode while the colloidal particles are retained. Thus the process of dialysis is triggered. The water in the compartments is renewed frequently or continuously.

It is important to note that electrodialysis can be used only for the removal of electrolytic impurities and cannot be used if non-electrolytic impurities such as sugar, urea, etc. are present.

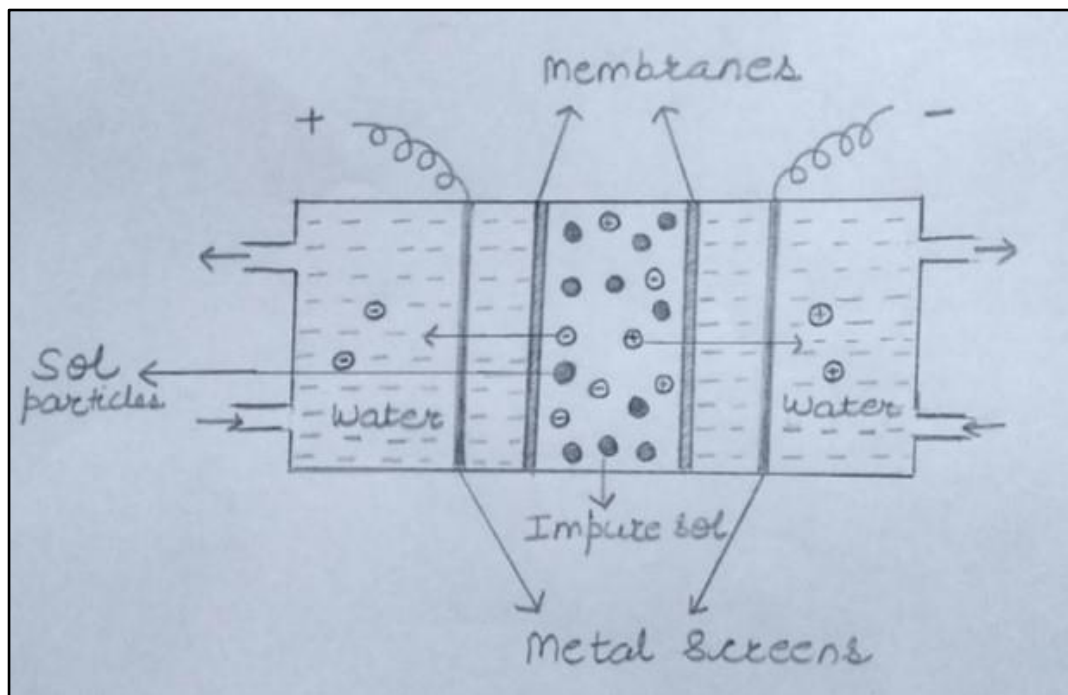


Figure 3.5: Electrodialysis

#### D. Ultra-filtration:

Ultra-filtration is a type of filtration through a semipermeable membrane in which separation is facilitated by pressure acting as the driving force. For purification of sols by this method, specially prepared filter papers called ultra-filters are employed.

The pore size of ordinary filter papers is larger than the size of colloidal particles. Hence if a sol is filtered through ordinary filter paper, the colloidal particles will also pass through it along with smaller sized solutes and solvent. However the size of these pores can be reduced by soaking the filter papers in a solution of gelatin or collodion (syrupy solution of 4% nitrocellulose in alcohol and ether) or a regenerated cellulose such as cellophane. The papers are then hardened by dipping them in formaldehyde solution and subsequently drying them. As a result the pore size of papers becomes small enough to hold back the colloidal particles. Such treated filter papers are called ultra-filters.

On filtering the impure sol through ultra-filters, the smaller sized impurity particles and solvent pass through it while the larger colloidal particles are retained on the ultra-filter paper. The colloidal particles on the paper can be redispersed into a fresh dispersion medium to obtain pure sol.

Filtration through ultra-filters is slow. To speed up the process, pressure is applied (either on the side of the sol or on the filtrate side by using a suction pump) to drive the filtration of solutes, hence the name ultra-filtration (Figure 3.6). The ultra-filters are quite delicate and fragile, therefore, they are usually supported on a wire mesh and then used.

It may also be noted that ultra-filters with different pore sizes can also be prepared by soaking the filter papers in solutions of collodion of different concentrations. By using a series of graded ultra-filters, not only the colloidal solution can be purified, but it can also be separated into fractions having colloidal particles of different sizes.

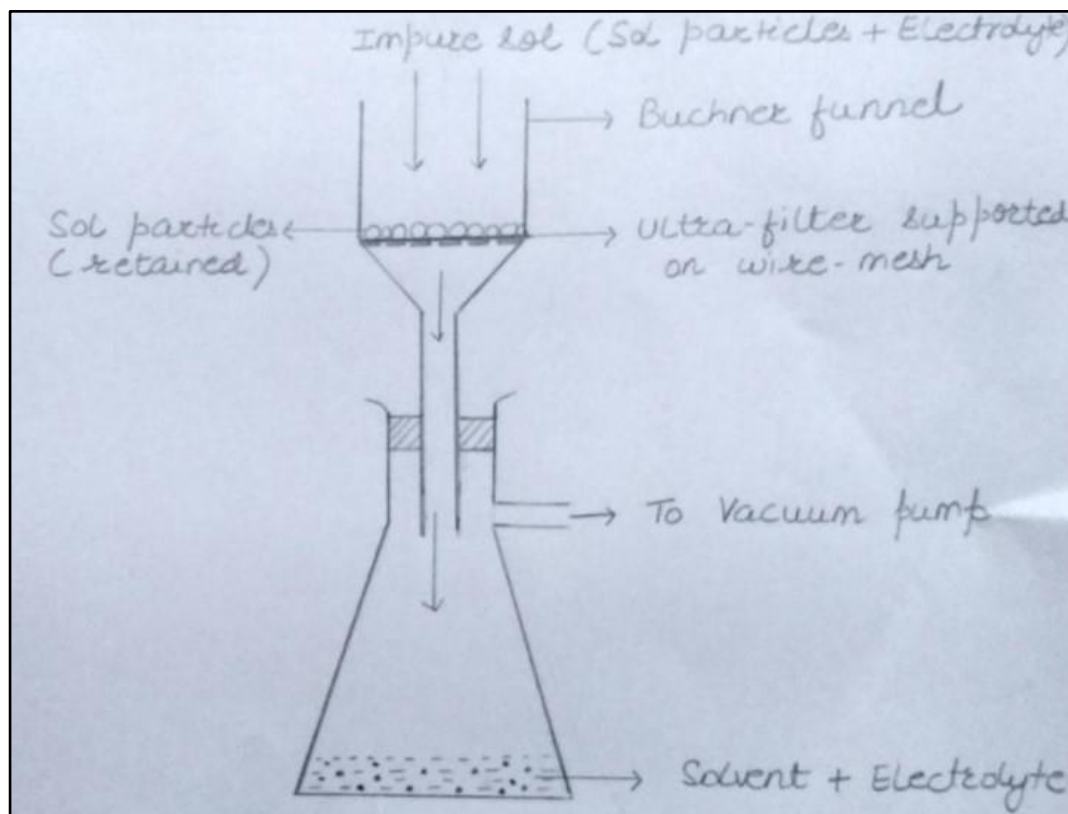


Figure 3.6: Ultra-filtration

### C. Ultracentrifugation:

The colloidal particles are always in a constant state of random and zigzag motion in the dispersion medium. Normally they remain suspended in the medium and the process of their settling down is very slow. But it can be enhanced by the application of centrifugal force.

Ultracentrifugation is a method of separating impurity particles from colloidal particles in the sol (according to the difference in their densities and / or particle size) by applying centrifugal force that is generated by spinning the colloidal solution around an axis (in a rotor) at a very high speed. The denser colloidal particles settle at the bottom while the lighter impurities remain suspended in the solution. In this purification process, the impure colloidal solution or sol is taken in a centrifuge tube and placed inside an ultracentrifuge. The rotor of the ultracentrifuge is spun at a very high speed (15000 revolutions per minute or more) about the central axis, thus generating the centrifugal force.



The colloidal particles being more dense, settle at the bottom of the tube and form the sediment while the impurities remain in the supernatant solution. This supernatant is called centrifugate (Figure 3.7).

The settled colloidal particles are removed from the tube and again dispersed into a fresh dispersion medium to get pure sol.

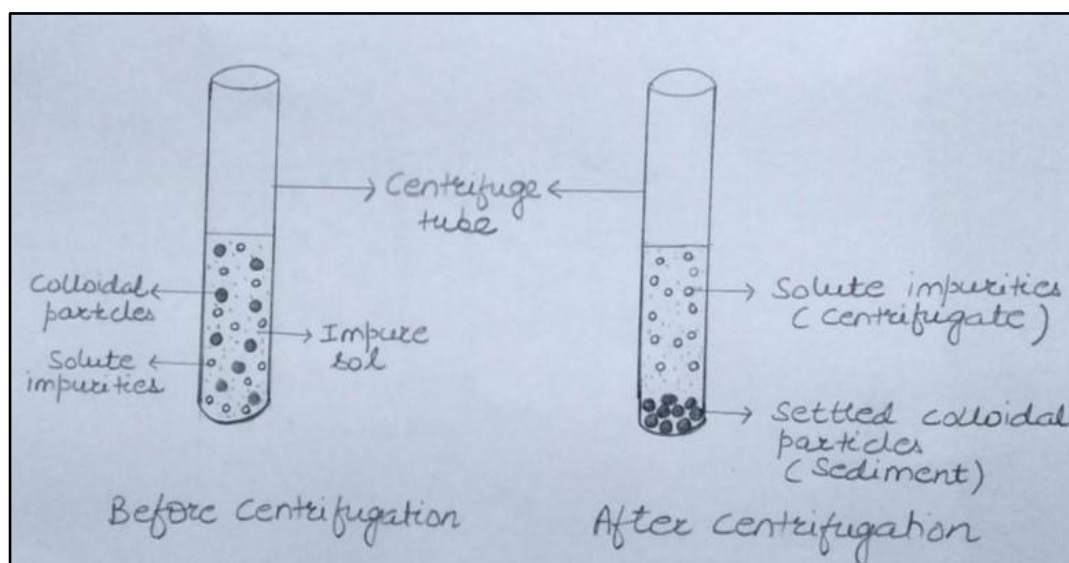


Figure 3.7: Ultracentrifugation of impure sol

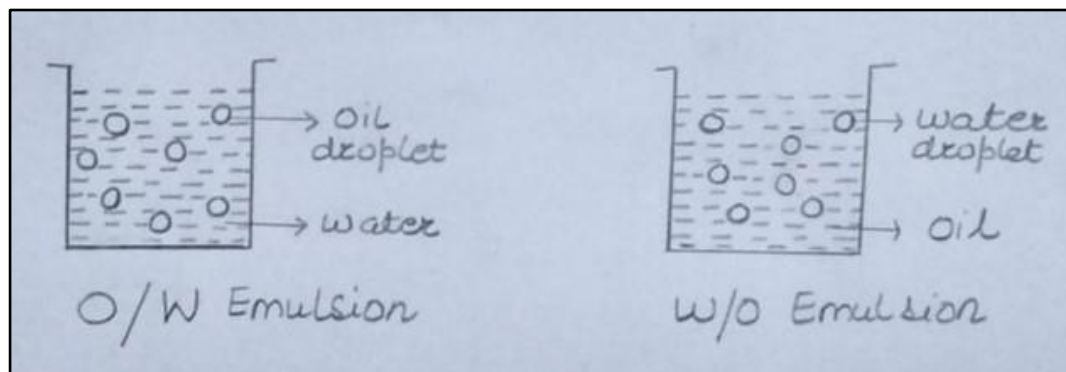
### 3.4 Emulsions:

Emulsions are colloidal systems in which both the dispersed phase and dispersion medium are liquids.

In an emulsion, small droplets of one liquid are dispersed in another immiscible liquid. Normally, one of the two liquids is water and the other liquid, which is insoluble in water, is called oil.

Accordingly, emulsions are of two types:

- a. **Oil- in –Water type (O/W type)** in which oil is the dispersed phase (internal or discontinuous phase) and water is the dispersion medium (external or continuous phase). For example- milk (with droplets of liquid fat dispersed in water), vanishing cream, etc.
- b. **Water- in – Oil type (W/O type)** in which water forms the dispersed phase and oil forms the continuous phase (dispersion medium). For example- cod liver oil, butter, etc.



**Figure 3.8: Two types of emulsions**

- **Emulsification and Emulsifiers:**

The process of dispersing one liquid in a second immiscible liquid, thus forming an emulsion, is called *emulsification*. An emulsion which is obtained by simply mixing and agitating together the two immiscible liquids is unstable. On standing, the liquids separate into two layers. To obtain a stable emulsion, it is essential to add a small amount of a third substance known as *emulsifier* or *emulsifying agent* or *stabilizing agent*. Emulsifiers are broadly of three types:-

- Certain long chain compounds with polar groups such as soaps, long chain sulphonic acids and sulphates.
- Lyophilic substances such as proteins, gum, agar-agar.
- Some insoluble powders such as lead sulphate, calcium carbonate, clay, carbon black, etc.

**Role of Emulsifiers:** Emulsifiers provide stability to an emulsion. They form a protective film around the droplets of the dispersed phase and thus stabilize the emulsion by reducing the surface tension at the oil-water interface. The protective barrier around the droplets keeps them separated and prevents their coalescence.

- **Preparation of Emulsions:**

Most emulsions are prepared by mixing the two liquids along with a suitable emulsifier and then agitating the mixture by passing it through a counter rotatory agitator or a homogenizer or a colloidal mill. One of the two liquids, as decided by the emulsifier, breaks down under the shearing forces to form droplets which get dispersed in the other liquid, thus forming an emulsion.

The type of emulsion obtained depends on the nature of the emulsifier used. According to the Bancroft's rule "*the phase in which the emulsifier is more soluble forms the continuous phase*". Hence water soluble emulsifiers tend to give O/W emulsions while oil soluble emulsifiers give W/O emulsions.

Griffin proposed a numerical scale, called Hydrophilic-lipophilic balance (HLB) which helps in predicting the type of emulsion that an emulsifier will produce. HLB is the balance of the size and strength of the hydrophilic and lipophilic moieties present on the emulsifier molecule. The HLB scale ranges from 0 - 20. If the HLB number of an emulsifier is in the range of 3.5 – 6 (indicating lipophilic nature), it is more suitable for use in W/O emulsion but if the HLB number is in the range of 8 -18 (indicating hydrophilic nature), the emulsifier is commonly used for O/W emulsions.

**Examples:** Acacia gum, whey protein are used as emulsifiers for O/W emulsions and emulsifiers such as lamp black (or soot), polyglyceryl oleate are used for W/O emulsions.

### **3.5 Gels:**

The colloidal system in which a liquid is dispersed in a solid is called gel. Some common examples of gel are: gelatin, jellies, fruit jams, curd, agar-agar, shoe-polish, silica gel, alumina gel, etc. It is observed that on cooling a warm solution of gelatin, it sets into a semi-solid mass (a gel). In the similar way, many lyophilic sols and some lyophobic sols of sufficiently high concentration, on coagulation under certain conditions, change into a semi-rigid and jelly like mass which encloses the whole liquid present in the sol.

*This process of transforming a sol into a gel is called gelation.* Some common lyophilic sols which convert into a gel are- gum arabic, agar-agar, starch, etc. Lyophobic sols that can undergo gelation are- silicic acid, ferric hydroxide, ferric phosphate sols, etc.

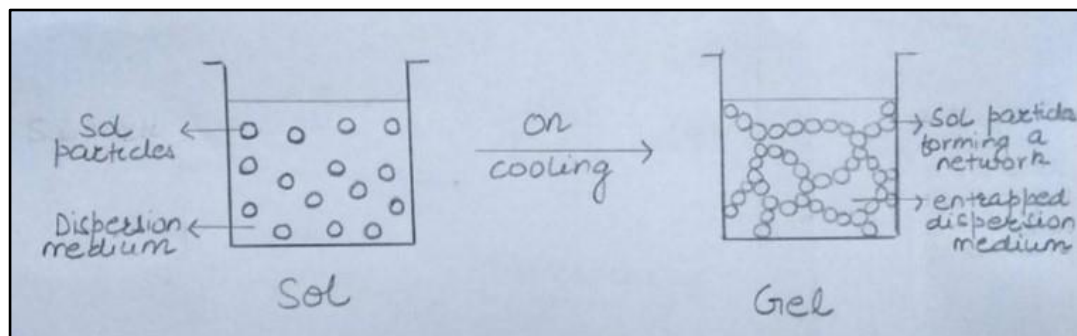
Based on their properties, gels have been divided into two categories:

- a. **Elastic Gels:** These gels possess the property of elasticity. On partial dehydration, they form an elastic solid which readily converts into the original form by addition of water followed by slight warming, if necessary. Examples of elastic gels are gelatin, agar-agar, starch, etc.
- b. **Non-Elastic Gels:** These gels do not possess the property of elasticity. On dehydration, they become glassy or change into a powder which cannot be converted back into the original gel by addition of water followed by warming. Silica gel, alumina gel are examples of non-elastic gels.

#### **A. Preparation of Gels:**

The following methods can be used for the preparation of gels:

- **By cooling sols of moderate concentration:** As discussed above, certain hydrophilic sols of moderate concentration, that have been prepared in hot water, change into a gel on cooling. In such sols, the particles are highly hydrated. On cooling, they come closer and cluster together to form larger aggregates which eventually form a semi-solid framework that encloses the entire dispersion liquid (Figure 3.9). This results in the formation of a semi - solid rigid mass with gel structure. For example- gels of gelatin, agar-agar, etc. can be prepared by this method.



**Figure 3.9: Formation of gel from sol**

- **By double decomposition:** This method is used to prepare hydrophobic gels such as silicic acid (silica gel) and aluminium hydroxide (alumina gel).

For preparation of silica gel, hydrochloric acid is added to an aqueous solution of sodium silicate. Highly hydrated silicic acid is precipitated which on standing changes into a gel.



Hydrated silicic acid

In the similar way, alumina gel can be prepared by mixing solutions of sodium hydroxide and aluminium chloride of suitable concentrations. As a result, an extensively hydrated precipitate of aluminium hydroxide is obtained which on standing sets into a gel.



Hydrated aluminium hydroxide

- **By change of solvents:** Certain hydrophobic gels have been prepared using this method. For example- When ethyl alcohol is rapidly added to an aqueous solution of calcium acetate of adequate concentration, the salt separates out to form a colloidal solution. When allowed to stand, it sets into a semi-rigid gel of calcium acetate enclosing the entire alcohol.