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PAPER - II

UNIT - II

DECOMPOSITION OF TECHNIQUES of ANALYSIS

Dissolution:-

⇒ The solute particles going into solvent to get a solution is called dissolution.

⇒ In a dissolution, the original nature of the sample doesn't changed.

⇒ By useing thermal methods and different conditions applied to the sample solution and to get original form of the sample. Hence, it is a dissolution.

eg: Starch solution.

Decomposition:

⇒ In this, the substances which cannot be dissolve are decomposed.

⇒ In a decomposition process, the original nature of the sample is completely changed.

⇒ Even applying thermal conditions, we doesn't get the original nature of the sample.

eg: Nacl solution.

Decomposition Techniques depends upon the following factors:

1) Polarity (or) Purity of Solvent
2) effect of Temperature
3) Hydrogen ion (H^+) Concentration
4) Complexing ability of anion
5) oxidising ability of anion/ion
6) solubility of salt with that Anion
7) Pressure

1) Polarity (or) Purity of Solvent :-

i) Solubility is depend upon the Polarity (or) Purity of Solvents

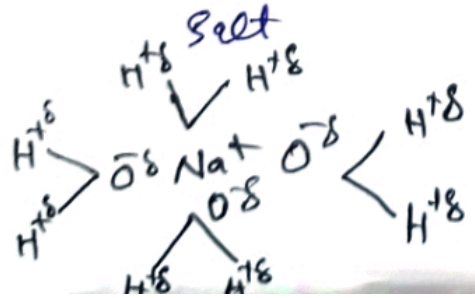
ii) Various Components are soluble in various Solvents

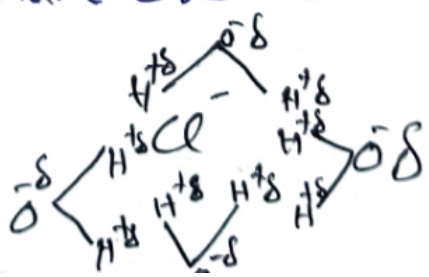
iii) In a decomposition technique, water is used as Solvent because it has high Polar nature than Compared to other Solvents.

For example NaCl is dissolved in water than to form Sodium chloride solution.

$$NaCl \xrightarrow{H_2O} Na^+ + Cl^-$$

Salt — Sodium chloride solution





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iv) In a sodium chloride solution, the water molecules should around the salt ions (Na^+ and Cl^-)

v) In case the concentration of sodium chloride, increases then also increasing of concentration of water.

Effect of Temperature :-

⇒ Solubility is depend upon the effect of Temperature

⇒ the Temperature is increases then the no. of monomers are also increases and no. of hydrogen ions are increased then the solute is soluble in solvent.

⇒ The temperature of H_2O, HCl, HNO_3, H_2SO_4 are

i.e

H_2O B.P ⟶ 100°C

HCl B.P ⟶ 110°C

HNO_3 B.P ⟶ 128°C

H_2SO_4 B.P ⟶ 338°C

Hydrogen (H^+) ion Concentration :-

⇒ Solubility is depend upon H^+ ion concentration.

⇒ If H^+ ion concentration increases then also Solubility increases.

⇒ The concentration of HCl, HNO_3 and H_2SO_4 are

HCl ⟶ 11.4N , HNO_3 ⟶ 16N , H_2SO_4 ⟶ 36N.

Complexing ability of Anion :-

⇒ Solubility is depend upon complexing ability of anion for example $CaCO_3$ insoluble in water and soluble in hydrochloric acid.

⇒ For example Silver Chloride is dissolved in ammonia then to form Silver ammonium Chloride complex.

$$AgCl \longrightarrow \left[Ag(NH_3)_2\right]^{+} Cl^{-}$$

Salt — Soluble complex

For example is HF (Hydrogen Fluoride), the fluoride ion ($F^{\ominus}$) is react with silica. Hydrogen fluoride is a suitable solvent for dissolving of silica (SiF_4)

Oxidising ability of Ion/Anion :-

⇒ Solubility is depends upon oxidising ability of anion.

⇒ Number of solvents used for dissolving of different salts.

⇒ In a decomposition technique, we are used the solvents like Nitric acid (NO_3^-), Sulphuric acid (SO_4^{2-}) and Perchloric acid (ClO_4^-).

(3)

⇒ Most of the salts dissolve in a perchloric acid than compared to Nitric acid and Sulphuric acid. But it is not prefer for a dissolving solvents why because it has highly exposing nature than compair to remaining solvents. So that, mostly Nitric acid is prefered for a decomposition of various samples.

Solubility of salt with that anion:-

⇒ At high concentration and high temperature of the solvents not preferable for solubility of salts.

⇒ Sulphuric acid have a high temperature like 338°C and high concentration 36N. So that it is not used as solvent in a decomposition technique because it to form a precipitation some of the salts like Ba, Ca^{+2}, Sb^{+4} etc--

Pressure:-

If pressure is increases the number of monomers are decreases similarly the solubility also decreases.

Nature of vessels:-

⇒ A suitable vessels is prefered for decomposition of samples.

⇒ If we are not used a suitable vessel is "corrosion" with solvents like acids than we produce bad results. So that we are select a suitable vessel.

Mostly glass vessel is used for decomposition of samples.

Borosilic:- which is with stand upto 800°C.

Quartz glass:- which is with stand upto 1200°C.

Glass carbon:- It is made up of by useing graphite and it is decomposition of sample at 500°C.

Polymers:- Polymers are also used for decomposition of sample.

Polyethylene:- which is with stand upto 600°C

Poly Propylene:- which is with stand upto 130°C

Teflon:- which is with stand upto 350°C.

Metal vessels:-

Metal vessels are also used for decomposition of some of the samples. In a metal vessel Nickel and Chromium are not used as

(+)

vessels why because these are corrosion. So that mostly crucibles are selected for decomposition of sample.

Principle of Ultrasonic decomposition :-

⇒ Now-a-days ultra sonic energy is required for decomposition of sample in various industr

⇒ The frequency of ultrasonic energy is higher than "Human audible range" (1-16 kHz)

⇒ when sound waves are induced in a molecular vibration of molecules solid liquid and gases substance.

⇒ when the energy is passes through a sample than it "expansion" and "compression" are takes place between the molecules.

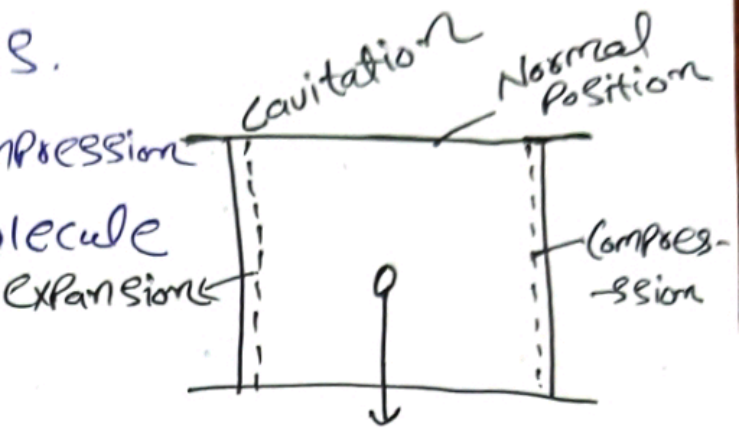
In this expansion means "Pull of the molecule" and compression means 'Pushes of the molecule.'

In a compression the number of molecules are come close together and repulsion forces are exist between the molecules.

In a expansion the no. of molecules are go away and attraction are takes place

between the molecules.

⇒ The expansion and compression are takes place in a molecule is known as "cavity".



⇒ The sample maybe containing water bubbles it is bombarted and is known as "Cavitation".

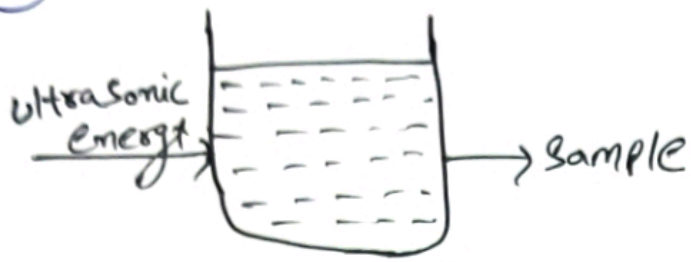
⇒ The whole process is takes place after the completion of decomposition of sample is "400 micro sec".

⇒ Ultrasonic decomposition have a two important applicatione.

1) ultra sonic both
2) ultra sonic probe (investigate throughly)

⇒ ultra sonic probe is more advantage application than compair to ultrasonic both. because in this Technique energy is directly focus into the sample. So that it required less energy and analysis of the sample in a less time.

(5)



Ultra sonic both

Ultra Sonic Probe

⇒ The "sonication time" required of a ultra sonic probe is always less than ultra sonic both. Mostly this technique is used for effective separation of samples.

Principle of decomposition by microwave energy:

⇒ Microwave ovens are rapidly used in various industries for efficient drying of acid decomposition of sample.

⇒ Micro wave energy is lies between IR radiation and electric field of electro magnetic radiation.

⇒ The frequency of microwave energy is 300 - 3,00,000 Hz.

⇒ Microwave energy is passes through a molecule and it is absorbed and the no. of ions in a sample effected in two ways.

1) Dipole radiation
2) Ionic Conduction

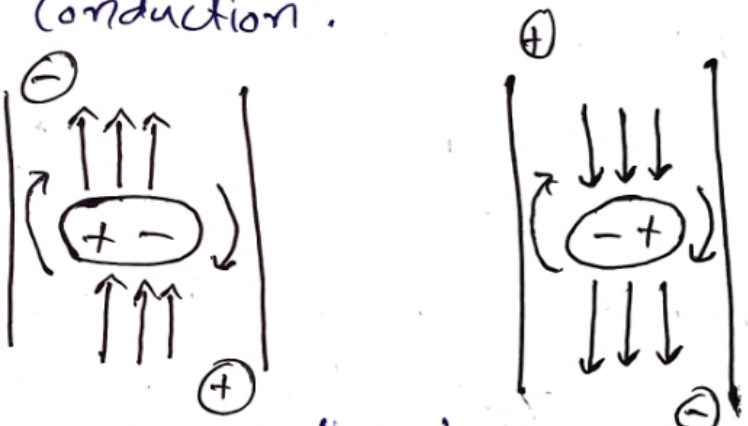
Dipole rotation :-

when a electric field is passes through a sample than the interaction takes place between no. of ions and these ions are rotated in a sample is known as "Dipole rotation". The phenomena is known as "Dipole moment".

Ionic Conduction :-

when a electric field is passes through a sample, In this no. of ions are migrated one position to another position is known as "Ionic Conduction".

Alignment (Adjusting) of a molecule in a Electric Field.



⇒ when a electric field is enter into sample the collision takes place between no. of ions in this kinetic energy is converted into thermal energy.

⇒ Required amount of energy is used for analysis of sample and loss of energy is calculated by using the following equation.

(6)

$$\text{Tan}\delta = \frac{\epsilon''}{\epsilon'}$$

Here, ϵ'' ⟶ di electric loss

ϵ' ⟶ di electric constant

⇒ "Domestic ovens" are used for drying of acid decomposition of sample.

⇒ In a acid decomposition sample is in various industries.

⇒ Now-a-days microwave ovens are used for efficient drying of sample.

⇒ Microwave ovens are more advantage than compared to domestic ovens due to these are producing simultaneously temperatures, moisture, Pressure and self generated energy.

⇒ Now-a-days by useing this technique twelve (12) samplee at a time analysed.

Re-crystallization:-

⇒ This technique is not applicable the substance react with the water.

⇒ In the organic reaction, the formation of product having melting point and boiling point. these are doesn't sharp melting point & boiling point than compared to original sample.

⇒ The satisfactory physical constants need to be for identification of purity of sample.

⇒ Distillation method is used for identification of purity of liquids (By solation).

⇒ Recrystalisation method is used for identificatio of purity of sample (solids).

⇒ Recrystals are formed in a cool condition & hot conditions. A solid sample is dissolve in a solvent and to form a solution. The containing solution is boiled and observe crystals than compaired to cool solutions, so that, more recrystals are formed in hot solution than compared to cool solutions.

⇒ For recrystalisation, hot soluents are prefered than compaired to cool soluents.

⇒ A solid sample dissolve in a suitable solvent and the containing solution is boiled

(7)

for maintainence of boiling point for formation of xecrystals and identification of purity of sample.

⇒ Non-Polar Compounds examplee " hydrocarbons are completely soluble in non-polar solvents Example hexane and ether.

⇒ Polar Compounds like hydroxyl groups (-OH, $-NH_2$, -(OOH) are soluble in Polar solvents alcohols.

⇒ No single solvent is not used for completly soluble of " binary mixtures".

⇒ No. of solvents are selected for solubility of no. of mixture of samples.

⇒ For identification of xecrystals in a binary mixture and apply different methods as below

Binary mixture is taken and add it first solvent the containing sample is mix well. then to form solution and it is boiled some time then add it second solvent until to get a "turbidity". Then also first solution is added drop by drop with swelling and till to form

a clear solution. No recrystals are identified in a clear solution then we are added "seed crystals", For identification of recrystals, If is also doesn't form recrystals. Then the containing solution is transfer from one flask to another flask and scrath with by useing glass rod it is also failed (Not observed any recrystals) Then the containing solution is boil sometime and it is placed under "dry ice Acetone bath". Then it is filter by useing "Buchner funnel" and identification of recrystals.

Hence, it is a recrystallisation method.

Decomposition of sample by Fusion:-

⇒ Fusion method is used for decomposition of different samples, which samples are directly doesn't dissolved in acids.

⇒ In a fusion technique flux material is used for decomposition of different samples and not dissolved in acids.

(8)

⇒ Fusion method need not necessary for dissolving of water soluble components, in a fusion technique no. of flux materials are used mostly sodium carbonate is preferred as a flux material for decomposition of samples in Fusion technique.

⇒ The melting point of sodium carbonate is 851°C and in sodium carbonate sodium is as interfer with other samples.

In a fusion technique the used flux material (sodium carbonate Na_2CO_3) is selected for fast decomposition of sample without contamination.

⇒ Fusion technique is applied at high quantity of sample and used high temperature.

⇒ This technique is also used for identification of components in a given sample.

⇒ In a fusion technique platinum crusible is used for analysis of samples.

Procedure:-

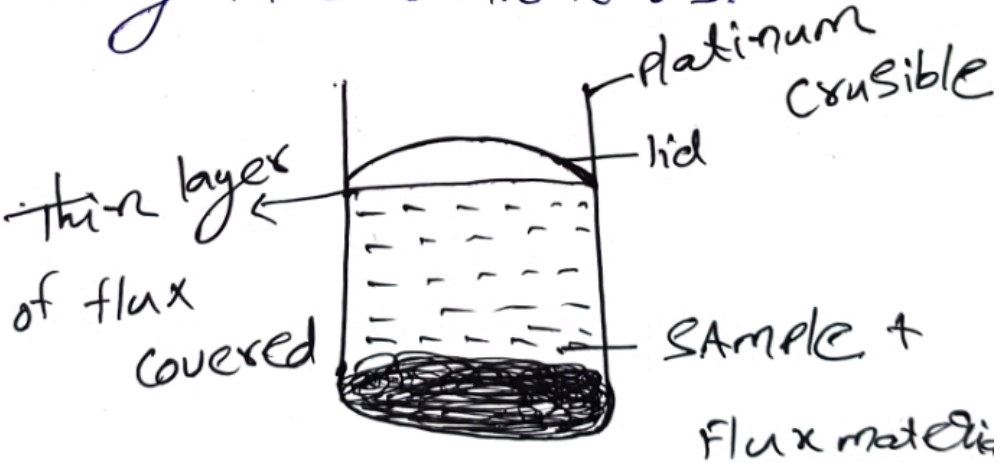
⇒ Let take 3-10gms of finally powder form of sodium carbonate and add it 0.5-1.0 gm of sample. The containing sample is taken

in a Platinum crusible and it is closed with a lid.

⇒ It is a heat upto 300°c for elimination of water molecules then it is cooled some time and it is again heated evaporation of CO_2 then the containing sample is to become melt form.

⇒ It is dissolve in suitable solvents and need not use hydrochloric acid because it is oxidised the components in a sample.

⇒ The containing sample is exhibit yellow colour, the sample containing "chromate" and the sample exhibit greenish blue colour. it containing manganese components.



$$M.SiO_2 + Na_2CO_3 \xrightarrow{\Delta} Na_2SiO_2 + M.CO_3$$

metal silicates — soluble — insoluble

$$\downarrow \Delta$$

$$M^+ + CO_2\uparrow$$

Fusion Techniques :-

These are Four types.

1) Alkali fusion :- Na_2CO_3, NaOH

2) Acidic fusion :- $NaHSO_4$, $Na_2S_2O_7$
$KHSO_4$, $K_2S_2O_7$

3) oxidation fusion :- Na_2O_2

4) Reductive fusion :- $Na_2CO_3 + Na_4B_4O_4$

Reductive fusion :-

⇒ In a Reductive fusion, sodium carbonates and sodium borates used as flux material.

⇒ Reductive fusion is used for the decomposition of resistance minerals.

⇒ The sample is fused in the Presence of flux and reducing agent is added.

⇒ The Components to be analised reducing in the lower valance state. then it is easily dissolve in water and (or) mineral acids.

⇒ oxides of heavy metals like as Lead (Pb), Antimony (Sb), Copper (Cu), Bismath (Bi) are easily

reduced this property is mainly used for the decomposition of precious metals like silver (Ag) gold (Au) and Telrium (Te) etc--

⇒ The procedure is divided into three stages.

Stage -1 :-

First the sample is mixed with flux material and it is fused in a crusible of fireclay in an electric (or) coneforced. The sample is partially fused then we add lead oxide which was fused the reducing of valancy state of given sample. By useing monoxides, carbonates, borax and lead oxides decompose the aluminium silicatee present to the respectiv silicates. The reduced lead circulate in the melt reducing silver, gold Tellurides to the metals. when we increase the temperature to form a "lead beads" in the bottom of the crusible The formation of lead beads is known as "regules", the crusible is remove from the electrical fireclay and allowed to cool.

Stage - 2 :-

The second stage is called as "slagging". In this technique slagging disk is used for decomposition of sample. The formation of regules (lead beads) is taken in a slagging dish and added borax mixture and the containing sample is heat upto 950°C, then to form 'borax slag'. In the process lead oxide is converted lead monoxide except Preceias metals.

Stage - 3 :-

In the final stage the regules weight is calculated and it is again heated until volitised of lead monoxide. Silver and gold remain in the form of granules is removed and it is carefully cleaned the content of Preccious metals is dissolved.

* Decomposition of sample by fusion with oxidation fusion :- Na_2O_2

⇒ The melting point of sodium peroxide is more than 500°C.

⇒ Sodium Peroxide can be melted by itself without decomposition and without the dangerous of an explosive.

⇒ At red heat, Sodium Peroxide decomposes the liberate oxygen it reacts explosively with substance that are easily oxidised ore, aluminium dust, sulphar etc--

⇒ Fusion Sodium Peroxide, is suitable for the decomposition of oresand Rocks.

⇒ In the determination of Sulphur, Tin, Tongestun(w), molibdinium (Mo), Vanidium (v) and Chromium (Cr),

⇒ The cases of fusion alcoholic salts of the highest oxidation state from these elements they are easily soluble in water and may be seperate by filteration from the insoluble hydroxides, Sodium Peroxide is used for decomposition of minerals and ores of Arsenate

⇒ In the Aqueos extract Arsenate may be seperated by reduction (or) by distillation.

⇒ The decomposition of silicate by fusion with sodium peroxide is used only if the rock to be analysed containing sulphides and Arsenides etc--

⇒ Frequently the metal is used for photometric determination of vanadium and chromium.

* Decomposition of sample by Acidic fusion:-

(or)

Decomposition of sample by fusion with sodium hydrogen sulphate (or) pyrosulphate.

⇒ Acidic fusion is a combination of the following components like → $NaHSO_4$ — M.P → 185°C

$KHSO_4$ — M.P → 219°C

$Na_2S_2O_7$ — M.P → 400°C

$K_2S_2O_7$ — M.P → 404°C

⇒ In a Acidic fusion, sodium hydrogen sulphate (or) pyrosulphates used for decomposition of various components.

→ Acidic fusion is essentially used for the decomposition of oxides.

⇒ Pyro sulphate, most useful flux material then compare to sodium hydrogen sulphate because it required less time for dissolution of sample.

⇒ Mostly in acidic fusion sodium hydrogen sulphate is also used for decomposition of sample.

⇒ Required amount of sodium hydrogen sulphate is taken and it is first converted to pyro sulphates then it is used for decomposition of various samples.

⇒ Accurate amount of sodium hydrogen sulphate is taken in a platinum (or) quartz crucible then add it small amount of water and it is heated until to form "foams (or) spatters". In the process by increasing temperature then water is liberated and the containing sample is converted to "melt" form.

⇒ Then required amount of sample is added to melt form substance the whole substance is again heated until to form

liquid (dark blue). the liquid form of substance is diluted with water (or) dil. H_2SO_4 (or) HCl. and the decomposition of sample is analysed.

⇒ Acidic fusion is also used for decomposition of Titanium and Boxides etc.. (Bauxite)

⇒ Acidic fusion is also used for fluorides

⇒ It is also used for decomposition of backlites. (Bakelite)

⇒ In the method of Acidic fusion, the sample may be completely not soluble even on applying all techniques then it is filter and seperate insoluble components and remaining components are decomposed and analysied.

Decomposition of sample by Alkali fusion :

(or)

Decomposition of sample by fusion with Carbonates :- (Na_2CO_3)

⇒ the melting point of sodium carbonate is 186°C.

Potassium carbonates (K_2CO_3) melting point is 984°C.

Sodium bicarbonates ($NaHCO_3$) M.P is 500°C.

Potassium bicarbonates ($KHCO_3$) M.P is 850°C.

⇒ Sodium carbonate generally contains impurities which may be effected any determination.

⇒ Purity of the substance to be analysed by using of major substance need not required for purity and minor substance to be required for pure.

⇒ Sodium carbonates may not be pure, therefore it is not sufficient to analyse large quantity of "trace elements.

↓ small quantity (Cobalt, copper, Fluorine, Iodine, Iron)

⇒ oxidative carbonate fusion is done in Platinum crusible in atmosphere of CO_2.

⇒ Anhydrous carbonates are used for decomposition of no. of minerals and silicate rocks.

⇒ For oxidative decomposition the amount of substance should not be to large. But in

the case of rocks with oxides decomposed by useing flux around 15 times high.

⇒ In fusion method based on the temperature, the nature of rocks decomposed, In these case of silicate rocks the temperature lies between 300-400°c. and oxidative rocks the temperature lies between 700-900°c.

⇒ Berillum minerals is also decomposed by using sodium carbonates.

⇒ Zirconium is also decomposed by useing sodium carbonates.

⇒ By useing Alkali fusion titanium is also decomposed.

⇒ In the case aluminium minerals react with borax and decomposed with by useing sodium carbonate at high concentration of aluminium.

⇒ Anhydrous sodium carbonates is also used, for decomposition of silicate rocks and minerals.

Decomposition of Sample by Sintering method:-

⇒ Sintering method is a suitable for decomposition of no. of samples.

⇒ In a sintering process, low quantity of sample decomposed at low temperature with in short period of time.

⇒ In a fusion technique, high quantity of sample is decompose at high temperature.

⇒ In a fusion technique, one problem is arised when high quantity of sample may be decompose at high temperature in a crusible. The sample may be corrosion with crusible at high temperature. In these case the sample is accurately not decomposed. In this technique some of the sample is also contaminated.

so that sintering technique is most preferable for decomposition of sample than compaired to fusion technique.

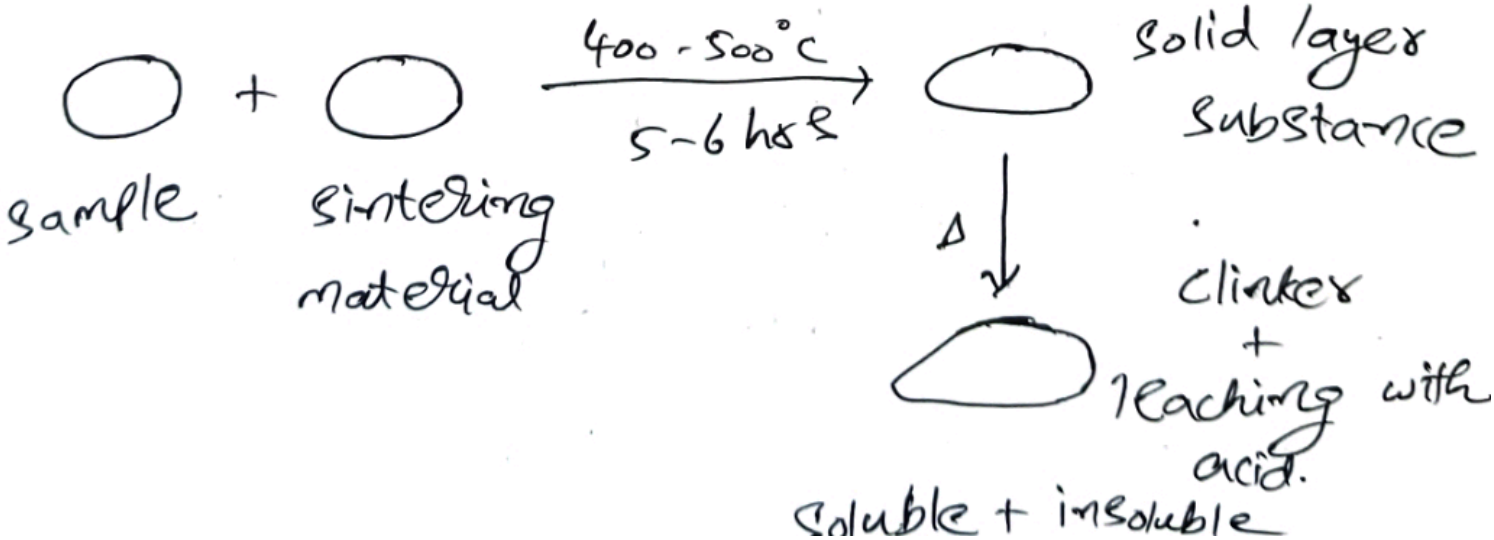
⇒ In a fusion method high quantity of sample decompose 5-15 times at 700-900°c in a

sintering technique. the sample is decomposed at 400-600°c the process is complete within 5-6 hrs.

Procedure :-

Required amount of sample is taken and add it required amount of sintering material. the containing mixture is taken in a crucible and it is heat upto 400-500°c within 5-6 hrs. Then it is converted to solid layer substance. the temperature is again rise, then it is also converted to one layer of sample is known as "clinker".

the clinker substance is leached with acid and seperation of soluble and insoluble components.



Decomposition of sample by Sintering with Sodium Carbonate :- (Na_2CO_3)

⇒ In sintering method sodium carbonate is also used for decomposition of no. of minerals, rocks and ores.

⇒ Sodium carbonate is also used for decomposition of quantitatively oxidised sulphites and crystals of potassium nitrite.

⇒ Lyme stone is also decomposed by useing sodium carbonate. In a sintering material (Na_2CO_3) is most preferable for decomposition of lymstone than compare to alkali fusion.

⇒ Sodium carbonate is also used for decomposition of "selinides" and "Tellurides".

⇒ In a sintering technique the given sample is decomposed in a crusible.

⇒ Commonly two types of crusibles are used in sintering technique they are Platinum crusible and Nickel crusible.

⇒ In a sintering technique most of the

(15)

Samples are decomposed in Nickel crusible because these crusibles are have a low cost, easily avaliable, at different temperature. not corrosion with samples.

⇒ Sodium carbonates is also used for decomposition of no. of oxides.

* Decomposition of sample by sintering with sodium peroxide :- (Na_2O_2)

⇒ Sodium peroxide is also used for decomposition of sample. it is used as the sintering mixture for analysis of silicate rocks.

⇒ The majority of minerals may be decompose by sintering with sodium peroxide in platinum crusible.

⇒ Platinum crusible is not prefer for decomposition of silicates by sodium peroxide because these crusible are easily corrosion with silicones.

⇒ As long as the sample contained no sulphides and the temperature apply over than 500°C.

⇒ This method is used for decomposition of no. of minerals, rocks and ores.

eg: Magnatite, Zipsum, Titanate, Boxite & quartz, graphite.

$$SiO_2 + 2Na_2O_2 \longrightarrow Na_2SiO_4 + O_2$$

$$TiO_2 + 2Na_2O_2 \longrightarrow Na_2TiO_4 + O_2$$

$$Al_2Be_3Si_6O_{18} + 16Na_2O_2 \longrightarrow 6NO_4SiO_4 + 3NaBeO_2 + 2NaAlO_2 + SO_2$$

$$2(FeO(Cr_2O_3)) + 7Na_2O_2 \longrightarrow 2Na_3FeO_3 + 4Na_2CrO_4$$

The liberated oxygen is corroded with suggested with reaction.

Q) difference between Fusion & sintering methods

Fusion	Sintering
1) Sodium carbonate, sodium hydroxide, sodium peroxide are used as Flux material.	1) Sodium carbonate, Sodium peroxide used as sintering material.
2) In these we are	2) In these, we are

(16)

a pure fusion mixture.

3) Time of reaction is 13-16 min

4) Time of interaction is taken as large time.

5) Commonly Platinum iridium crusibles are used.

6) Partical size is large than compare to sintering.

7) Corrosion of vessel is very high

8) The liquid formation of fusion is response for decomposition.

9) The sample is mix with 10-15 times and heat upto 900-1200°C

10) In the fusion technique the sample is not easily decompose than compare to sintering.

used a pure flux substance.

3) Time of reaction is 5-6 hrs

4) Time of interaction is taken as less time.

5) In this Platinum, iridium Nickel crusibles are used.

6) Partical size is smalle than compare to Fusion.

7) Corrosion of vessel is very less.

8) The formation of sintering compound is called clinker.

9) The sample is mix with 2-3 times and heat upto 400-600°C.

10) In the sintering technique the sample is easily decompose than compare to fusion.

ii) In fusion technique high quantity of sample and high temperature is used for decomposition.

ii) In sintering technique low quantity of sample and low temperature is used for decomposition.

Decomposition of Sample with organic Solvents:

⇒ organic solvents used for decomposition of no, of samples. This solvents also used for isolation of decomposition samples.

⇒ three types of organic solvents are used for decomposition of samples. They are

1) Acetic acid
2) oxalic acid
3) Tartaric acid

Acetic acid :-

A 10% of solution used for decomposition of mixture of Calcium and flourides, And these are digested is carried out in a waterbath. The besides Calcium Carbonates, other Carbonates and Calcium Sulphates also decomposed by Acetic acid.

(17)

May be these are Partially soluble in these chlorides and sulphides, must be oxidised with a solution of 10% Bromin in glacial acetic acid. 5% acetic acid used for decomposition of silicates.

eg:- zinc silicate.

2) oxalic acid :-

⇒ scheelite (used as ore of tungsten) dissolve easily hot 10% oxalic acid solution. In this insoluble residue containing of calcium oxalate.

⇒ oxides of oxalic acid used for dissolution of Pyrolusite.

⇒ The sample is decomposed with a amount of 0.2 moles oxalic acid in H_2SO_4 medium and any excess acid is determined by oxidimetric titration after boil it for 30min.

3) Tartaric acid :-

Tartaric acid is used for analysis of antimony ores and take antimony ores is dissolved in Con. Hcl and add it tartaric acid before dilution with water.

To avail the hydrolysis of antimony. Tartaric acid is used dissolved oxygen content. the antimony minerals and alloys also decomposed by nitric acid and tartaric acid.

Eg: Naturally available Alloyes.

* Decomposition of Sample by Sintering with Sodium Peroxide.

⇒ The melting point of Sodium Peroxide is 495°C.

⇒ Highly resistance minerals can be decomposed by useing Sodium Peroxide effectively at low temperature.

⇒ It is a function of both decomposing agents and oxidant.

⇒ Platinum Crusible can be used for the Sintering.

⇒ A mixture of Soda and lithium Carbonates (2:1) melting at 470-480°c has been used to decomposition.

⇒ Sodium Carbonate Partially decomposed at 900°c

$$Na_2CO_3 \rightleftharpoons Na_2O + CO_2\uparrow$$

In this sodium oxides react with sample and decompose at high temperature to occur corrosion with crusible, so that to maintain low temperature for decomposition of sample with sodium peroxide in a crusible.

* Decomposition of sample by fusion with Alkali hydroxyl group :- (NaOH, KOH)

⇒ Sodium hydroxide and potassium hydroxide is also used for decomposition of various samples.

⇒ Pure sodium hydroxide melt at 320°c but at 10 - 20°c. lower depends upon the nature and impurities.

⇒ It is strong basic compounds with sodium carbonates.

⇒ Potassium hydroxide melting point is 360°c.

⇒ Potassium hydroxide melting point is greater than sodium hydroxide.

⇒ The Fusion with hydroxide is generally used to decomposition of quartz silicates, sand, clay, carbides of various metals.

⇒ Teflan can be also used with the fusion temperature at 2300°C.

⇒ Natural oxides of tangestate, titaey, chlorides in several metal oxides, flourides can also be decomposed easily with sodium hydroxide fusion mixture

⇒ Fusion of quartz with hydroxides mixture reacts with quickly and forming water soluble sodium silicate compounds.

⇒ Chromate can be decomposed by useing sodium hydroxide but in the process of decomposition can be completed by only after oxidising sodium peroxide used as oxidising agent.

⇒ The decomposition of phosphates, flourides are more greaterthan sodium carbonates.

⇒ Natural alkali earth sulphates also decomposed in molten hydroxides.

⇒ the melt form of substance is digested with 2% sodium carbonate solution and alkali earth carbonates are separated from the sulphates by filter.

→ The fusion is carried out with hydroxide in iron (or) Nickel (or) silver, gold but no. of metals taken in a platinum crusible.

* Decomposition of sample by useing inorganic solvent:-

1) Decomposition of sample by useing H_2SO_4.

1) H_2SO_4 B.P is 338°C
2) H_2SO_4 Normality is 36N
3) Con. H_2SO_4 is the hot condition act as mild oxidising agent.
4) H_2SO_4 is an efficient solvent for the decomposition of no. of minerals and ores.
5) H_2SO_4 at high temp is mostly used for decomposition of sulphide ores, Arsenic, antimony and Tin.
6) H_2SO_4 is also used for decomposition of no. of minerals like Scandium & Telurium.
7) The temperature of decomposition must be maintain below 100°c to avail voltalization Scandium salts.

8) Halides especially flouride ion are easily decomposed by H_2SO_4.

9) No. of oxides also decomposed by useing H_2SO_4

10) It is used for the dissolution of Carbonates & Phosphates Solvents.

11) Natural minerals Contain Sulphites is also easily decomposed at high temp by useing H_2SO_4.

⇒ Mostly different Crucibles are used for the decomposition of Sample by H_2SO_4. But Platinum & gold vessels are not used because these are Carroded with acids.

⇒ upto 250°C gold is stable with respect to effective of 96% H_2SO_4.

⇒ Poly ethylene is stable upto 50°C in H_2SO_4.

⇒ Teflon is stable upto 300°C in H_2SO_4.

2) Decomposition of Sample by $HClO_4$:-

⇒ Perchloric acid is a strong acid.

⇒ The B.P of Perchloric acid is 203°C.

(20)

⇒ No. of Compounds decomposed by $HClO_4$ except NH_4, k, Ce, Rb.

⇒ The following compounds ammonium, Potassium Ce, Rb, rapidly soluble in water and other organic solvents.

⇒ Hydroxy solvents are Ethanol, Cellulose, Polyhydroxy alcohols also decomposed with Perchloric acid and increasing exposing, nature by useing lead sulphate then control the explosing nature of $HClO_4$.

⇒ The sulphides of bismuth and Chlorides are also decomposed by Perchloric acid then to form bismuth Perchloride. it is a useful solven

⇒ Phosphate is not dissolve in $HClO_4$, then to add Nitric acid and to form complex then decomposed.

⇒ Perchloric acid is a strong oxidising agent and used as dehydrating agent.

⇒ No. of monozites are easily decomposed by Perchloric acid.

⇒ No. of minerals and aluminium bridge minerals are easily decomposed by $HClO_4$.

⇒ the precipitation of silisilic acid (H_2SiO_3) by adding perchloric acid.

Decomposition of Sample by Nitric acid HNO_3

⇒ Nitric acid is avaliable in concentrate and dilution.

⇒ Boiling point of Nitric acid 128°c.

⇒ Normality of HNO_3 is 16N.

⇒ If sulphides, Arsenides and phosphates are also dissolved in HNO_3 then sulphide is converted into sulphate and Arscinide is converted arscinate.

⇒ It is generally stored in polyethylene (or) glass (or) Teflon vessel.

⇒ Gold, Platinum, uranium, Rhodium & iridium do not dissolve in Nitric acid and silver, lead are easily soluble in Nitric acid.

⇒ Majorities of metals are easily decomposed by Nitric acid except gold, Platinum, uranium, Rhodium & iridium.

⇒ For H_2O dissolution of no. of components either decomposition of Nitric acid.

⇒ By useing Nitric acid Copper ores are easily decomposed.

⇒ By analysis of metal ion can be carried out by useing volumetric and gravimetric technique, the presence of nitrates can be removed by repeated evaporation with Nitric acid.

⇒ No. of minerals like bismuth & Tin are easily decomposed by Concentrated Nitric acid.

⇒ Nitric acid is also used for decomposition of no. of components by useing "$HNO_3 + HCl$" "$HNO_3 + HF$" & "$HNO_3 + H_2SO_4$".

4) Decomposition of sample by Hydrochloric acid:

⇒ Hydrochloric acid is used for decomposition of no. of ores like iron.

⇒ The Boiling Point of Hcl is 110°c.

⇒ The Normality of Hcl is 11.4 N.

⇒ The decomposition with Hcl is generally carried out glass vessel.

⇒ If Platinum (or) gold vessel are used they may be corroded due to the prolonged contact with Hcl.

⇒ Dissolution of Heavy metals like Carbonates and flouro Carbonates is done by Hcl.

⇒ oxides of manganese ore is also soluable in Concentrated Hcl.

⇒ By useing hydrochloric acid no. of ores of Zink Barium, Calcium are dissolved in hydro-chloric acid.

⇒ Boron ores also decomposed by useing Hcl, at high temp under the reflux condition is eliminated the boric acid.

⇒ The salts of Phosphate, chlorides, hydroxy ions are also soluble in Hcl.

⇒ vanidium, Tungestate, Arscinate can also easily decomposed by Hcl.

⇒ No. of metals may be not dissolved in Hcl solvent then used for the Combination of Nitric acid and hydrochloric acid. ($HNo_3 + Hcl$)

used for the decomposition of that type of metals.

5) Decomposition of sample by hydrogen mono flouride (HF)

⇒ In dissociation of minerals and ores, hydrogen mono flouride has an effective greater than other mineral acids.

⇒ No. of minerals and ores also soluable in hydrogen flouride.

⇒ Hydrogen flouride is dissociate in very less extent in water and it forms hydrogen flouride ion.

⇒ The flouride ion complex with several metals like V, tungestan, zirconium & aluminium.

⇒ HF can be stored in Polyethylene (or) Teflon vessel.

⇒ The flouride of several metals especially the 4th group are highly volatile. The mineral must be decomposed impresence of H_2SO_4.

⇒ In the analysis of silicate the mixture

of

of hydrogen flouride and mineral acids are used for the decomposition of alkali metals.

⇒ The hydrogen flouride completely not decomposed and dissociate.

$$HF \longrightarrow H^+ + F^-$$

(soluble)

$$H^+ + F^- \longrightarrow HF^- \text{ (complex)}$$

⇒ Silica is taken and added HF then to Form " H_2SiF_6 ". Then also it converted into silicon tetra flouride (base medium) and HF.

$$SiO_2 + HF \longrightarrow H_2SiF_6 \longrightarrow SiF_4 + 2HF$$

⇒ The chalcopyrame is decomposed by useing a mixture of perchloric acid & Nitric acid.

⇒ Teflon beakers are used for decomposition of samples at 200°c in Presence of Perchloric acid & Nitric acid.

⇒ For the determination of Boran and Silicate in the Process to avaid. the

(17)

The formation of Boran triflouride (BF_3) by adding ortho phosphoric acid. It will be estimate the formation of Boran tri-flouride.

⇒ If we analysis Trace elements by adding hydrogen flouride and orthophosphoric acid (H_3PO_4). It is analysis by useing atomic absorption spectroscopy and indective coupled Plasma mass spectroscopy (ICPMS)

⇒ Hydro flouride is also used for the quanti-tative determination of uranium and morion.

⇒ No. of salts are easily decomposed in presence of mixture of hydrogen flouride and Nitric acid.

⇒ Nitric acid is easily evaporated then compare to chlorides. In HCl medium chlorides is also Precipitated some salts.