MODULE 5

MODULE 5

INSTRUMENTAL METHODS OF ANALYSIS AND NANOMATERIALS

INSTRUMENTAL METHODS OF ANALYSIS

Chemical analysis involves

- Identification of elements of a compound in a mixture is called qualitative analysis
- Determination of amount of element or substance present in the sample is called quantitative analysis.

Quantitative analysis involves

- Titrimetry, where the substance to be analyzed is made to react with a standard solution of appropriate reagent.
- Gravimerty, where the substance to be analyzed is precipitated by the addition of reagent, the precipitate is collected and weighed.
- Instrumental analysis, where the substance to be analyzed is converted into an electrical signal, that is measured using a suitable device.

Advantages of Instrumental methods of analysis:

- It is faster than chemical methods.
- Requires only small quantity of sample.
- Easy when a large number of samples have to be analyzed.
- Accurate results are obtained.

COLORIMETRY:

Electromagnetic radiation is a form of energy, travels in space at high velocity. Based on the wavelengths, electromagnetic radiations are divided into γ-rays, X-rays, UV rays, visible rays, infra red rays, micro waves and radio waves. These radiations when passed through matter, get absorbed and bring about various changes.

Therefore, when radiations like UV, IR and visible are passed through the sample, a part of it is absorbed, resulting in decrease in intensity of radiation. The wavelength of the radiation absorbed is a characteristic of that substance and quantity of radiation absorbed is a function of its concentration. Hence this principle is used to identify and quantify a substance. The Beer-Lambert law relates the light absorbed with the chemical analysis.

PRINCIPLE:

When a monochromatic radiation of intensity I is passed through a solution of sample under investigation, taken in a cell of thickness t, a portion of the radiation is absorbed (Ia), a portion is reflected, (Ir), remainder is transmitted (It).

Then, $I = I_a + I_r + I_t$

For glass cells, Ir is negligible.

Therefore, $I = I_a + I_t$.

LAMBERT'S LAW:

It relates incident, absorbed and transmitted light. "When a monochromatic light passes through a transparent medium, the rate of decrease in intensity with the thickness of the medium is proportional to intensity of light."

i.e., $\dfrac{-dI}{dt} = kI$,   k is proportionality constant.

i.e., $\dfrac{-dI}{I} = kdt$

Integrating between the limits $I = I_o$ when $t = 0$ and $I = I_t$ when $t = 1$,

$\ln\left(\dfrac{I_o}{I_t}\right) = kt$

Or, $I_t = I_o\, e^{-kt}$

This equation shows the decrease in intensity of transmitted light with increase in thickness of medium.

BEER'S LAW:

It states that "the intensity of the transmitted light decreases exponentially as the concentration of the medium increases arithmetically."

Then, $I_t = I_o\, e^{-kc}$, Where, c = molar concentration of sample solution.

Combining equations for Beer' law and Lambert's law, equation for Beer-Lambert's law is obtained and is written as,

$I_t = I_o\, e^{-kct}$ Or, $I_t = I_o\, 10^{-\varepsilon ct}$

Where ε is molar absorption coefficient, a constant for a given substance at a given wavelength. The unit is $dm^3 mol^{-1} cm^{-1}$. The above equation can be written as

$\log\left(\dfrac{I_o}{I_t}\right) = \Sigma\, ct.$ is called Beer - Lambert's law.

The term $\dfrac{I}{I_o}$ is called Optical Density or Absorbance A.

$\dfrac{I_t}{I_o} =$ Transmittance, T

Therefore relation between A, T and E is given by,

$A = \varepsilon\, c\, t$

$= \log\left(\dfrac{I_o}{I_t}\right) = \log\left(\dfrac{1}{T}\right) = -\log T$

2

If path length of the cell is kept constant, then absorbance A is proportional to concentration c.

ESTIMATION:

When Cu^{2+} ions are treated with ammonia solution, a deep blue colored cuprammonium complex is obtained. The absorbance is measured at 620nm (λmax) since the complex shows maximum absorbance at this wavelength. When absorbance of known solutions is plotted against concentration, a straight line is obtained. From this, unknown solution concentration can be obtained by measuring the absorbance of that solution.

$$Cu^{2+} + 4NH_3 \longrightarrow \left[Cu(NH_3)_4 \right]^{2+}$$
Deep blue
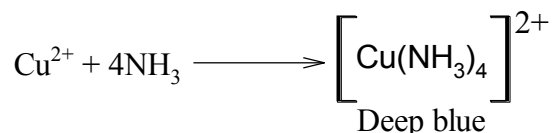
PROCEDURE:

5, 10, 15, 20 and 25ml of standard Copper sulphate solution is taken in separate standard flasks. 5ml of ammonia is added to each of these flasks and made up to the mark using distilled water. The flasks are stoppered and shaken well for uniform concentration. To the given test solution, 5ml of ammonia is added and made up to the mark with distilled water, stoppered and shaken well. A blank solution is also prepared taking 5ml of ammonia, making up to the mark using distilled water and shaken well. The absorbance of solutions against blank is measured at 620nm using colorimeter. A calibration curve is drawn by plotting concentration of Cu (mg/cm^3) against absorbance. From this, concentration of Cu in test solution can be determined.

The method is used to estimate Copper in brass, Mn in steel, glucose in fluids etc.
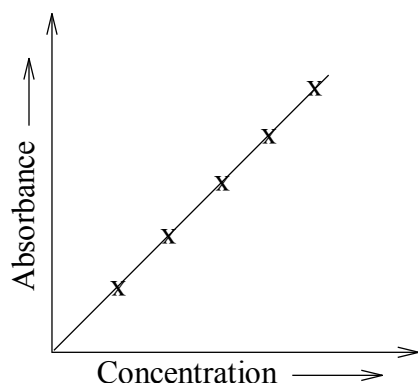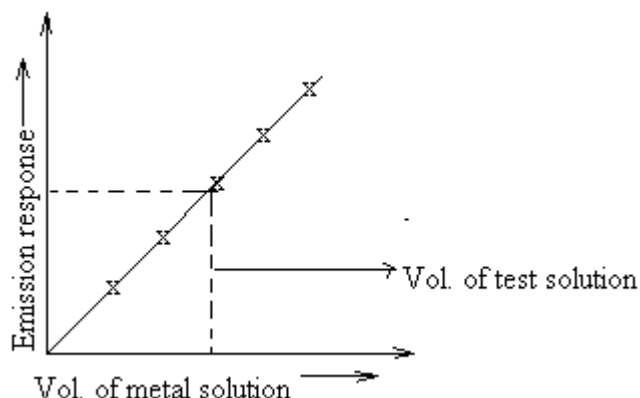

FLAME PHOTOMETRY:

Principle: When a solution containing a metallic compound of Na, K, Li, Ca and Ba is aspirated into a flame, a series of changes take place at the flame. First, the solvent gets evaporated leaving behind the salt in the flame. The salt then gets evaporated into vapors' of the salt, which further undergo dissociation into the constituent atoms. The metal atoms formed in the flame may absorb heat energy from the flame and get electronically excited to their higher energy level. As they are unstable at this excited level, the atoms fall back to their ground state by

emitting the energy difference between the excited and ground state in the form of light radiation. The intensity of the light radiation emitted is proportional to the number of atoms in the excited state, which in turn is proportional to the concentration of the solution fed into the flame. Hence by measuring the intensity of emitted radiation by a suitable device, the concentration can be measured.

Different metals emit their characteristic radiation at different wavelengths; they do not interfere with each other, even when they are present together.

Instrumentation:

Flame photometer consists of an automizer, mixing chamber, burner, filter, detector and a read out device. Pressurized air is passed into the automizer and the suction. It draws solution of the sample into the atomizer, where it mixes with the air stream as a fine mist and passes into the mixing chamber. In the mixing chamber, it mixes with fuel gas and then passes into the burner
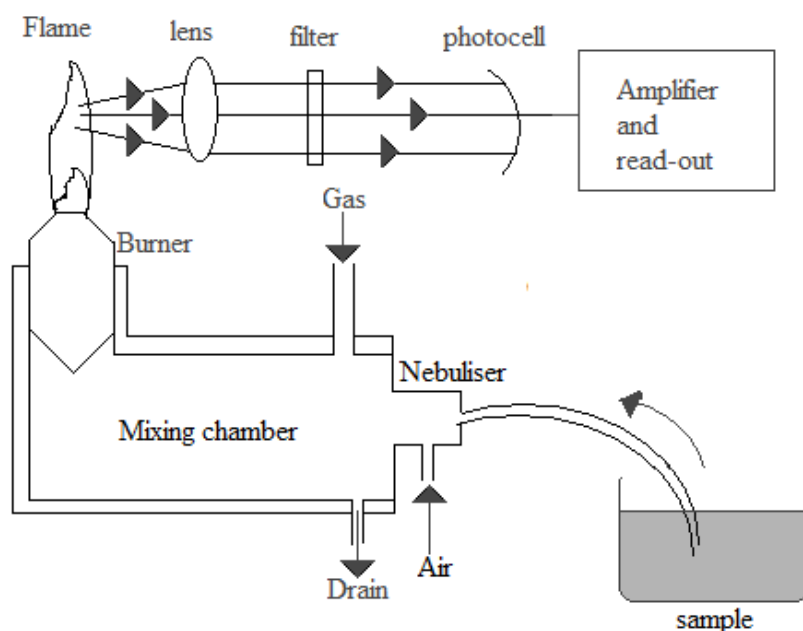
Where, the mixture is burnt. The radiation from the resulting flame passes through a lens and finally through a filter, which permits only the radiation characteristic of the element under

investigation to pass through the detector. The output from the detector is read out on a suitable read out system.

ATOMIC ABSORPTION SPECTROSCOPY

It is a technique for determining the concentration of a particular metal element in a sample.. Amount of energy absorbed gives the estimate of the concentration of analyte in the sample.

Theory:

In Atomic absorption spectroscopy atoms are excited by the flame. The excited atom absorbs light from the light source resulting in the net decrease in the intensity of beam. The amount of light absorbed is proportional to the concentration of the element.This follows Beer- Lambert law A= $\epsilon\, l\, c$

Where l is path length, A is absorbance, C is concentration of the solution, ϵ is molar absorption coefficient.

Instrumentation: Atomic absorption spectroscopy consists of the following components:

1. Hollow cathode lamp
2. Nebulizer
3. Atomizer
4. Monochromator
5. Detector and Recorder

Hollow cathode lamp: light source used is hollow cathode lamp. It set the instrument at certain wavelength suitable for certain element. Light source is placed in such a way that radiation passed through flame.

Nebulizer: it sucks up sample at control rate & creates fine spray for the introduction into the flame. The aerosol, fuel and oxidant mixed thoroughly for the introduction into the flame.

Atomizer: Sample when heated broken into atoms.

Monochromator: Used to select the specific wavelength of light absorbed by the sample.

Detector: Light selected by the monochromator is passed into detector it converts light signal into electrical signal.

Recorder: signal is recorded.
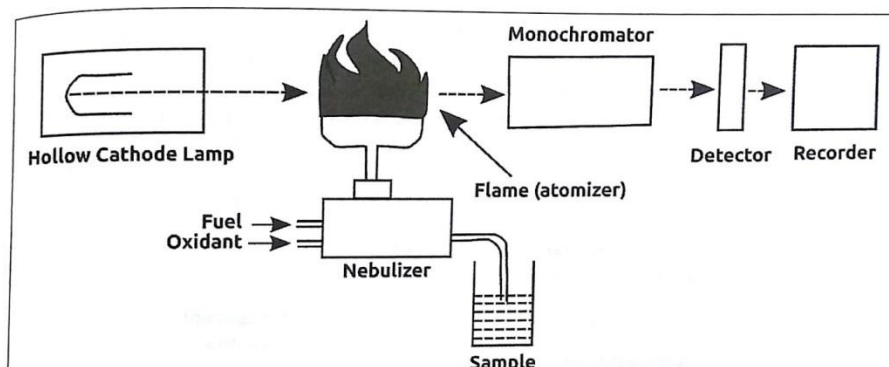
Applications:

1. Used for the quantitative measurement of metal elements in soil, plant, food industry.

2. Used for the analysis of metal ions in blood, saliva , urine sample like Na, K , Mg &Ca

3. To estimate lead in petroleum products.

4. To determine heavy metal like iron, zinc, lead , nickel mercury etc in environmental sample.

5. To determine metal concentration in ground water and bore well sampling before using drinking.

POTENTIOMETRY:

The concentration of ionic solution can be determined by measuring emf by the method of potentiometric titration.

PRINCIPLE:

The potential of an electrode is given by Nernst equation,

$$E = E^o + \frac{0.0591}{n} \log\left[M^{n+}\right]$$

i.e. The potential of an electrode depends on concentration of ion to which it is reversible. This method can be used in the determination of end points of acid-base titrations, red-ox titrations etc.

When a known volume of analyte is titrated with a standard solution, neutralization or red-ox reaction takes place. During the course of titration, concentration of product will be continuously altered. If a metal electrode, reversible with respect to corresponding ions is placed in the solution, the potential will vary throughout the titration, which can be determined using a suitable device. Initially, the change in potential is very small. At the equivalence point, when the amount of titrant added is equivalent to the amount of analyte present, there will be a sharp rise in potential. Beyond the equivalence point, there will be no significant change in potential. By plotting a graph of change in potential against the volume of titrant added, the equivalence point can be determined.

INSTRUMENTATION:

Potentiometer consists of a reference electrode, an indicator electrode, and a potential measuring device. The indicator electrode responds to the changes in concentration of analyte. A reference electrode like saturated calomel electrode is also used. A known volume of analyte is taken in the beaker and its potential is determined. The titrant is added in increments of 1ml and emf is measured each time.

At equivalence point, emf increases rapidly. At this point, titrant is added in small increments of 0.1ml. a few readings are taken beyond the end point. Thus the changes in potential at different volumes of titrant are recorded.
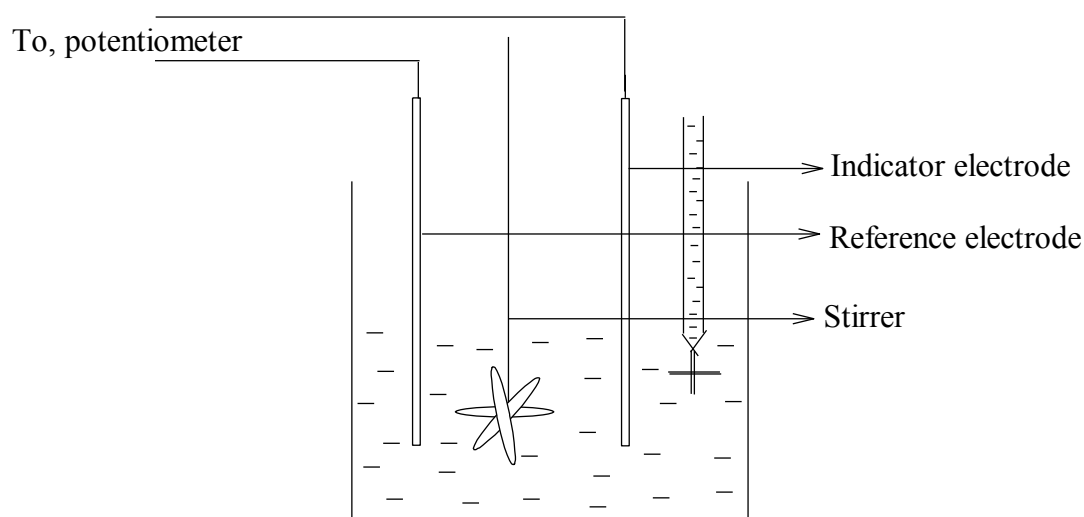
ADVANTAGES:

✓ Potentiometric titrations can be carried out in colored solutions where indicators cannot be used.

✓ The prior knowledge of relative strengths of acids and bases to select a proper indicator is necessary in normal titrations, whereas such knowledge is not required in potentiometric titrations.

✓ By potentiometry, it is possible to determine the end point in titrations of very weak acids or very weak bases.

Based on the nature of chemical reactions, potentiometric titrations are classified as

❖ Neutralization titrations

❖ Redox titrations

❖ Precipitation titrations

❖

1. Neutralization (Acid – Base) titrations:

If a solution of HCl is titrated against NaOH, an electrode whose potential depends on H^+ ion concentration (glass electrode) is placed in HCl and coupled with a reference electrode to form a galvanic cell. The emf of the cell is measured potentiometrically, and is given by

$E_{cell} = E_{ref} - 0.0591 \log[H^+]$

$\quad = E_{ref} + 0.0591 \, pH$

As the titration proceeds, H^+ ion concentration goes on decreasing (pH increases) and hence emf of the cell increases. Initially, emf increases slowly & then rapidly at the neutralization point. After the neutralization point, there is no significant change in emf. A graph of change in emf against volume of titrant is plotted and equivalence point is determined. The end point in acid-base titrations can also be found by direct measurement of pH of solution after each addition of a base to acid, using pH meter. The equivalence point is found by plotting a graph of emf against volume of a base, also by plotting $\Delta E / \Delta V$ or $\Delta^2 E / \Delta V^2$ against volume as follows.
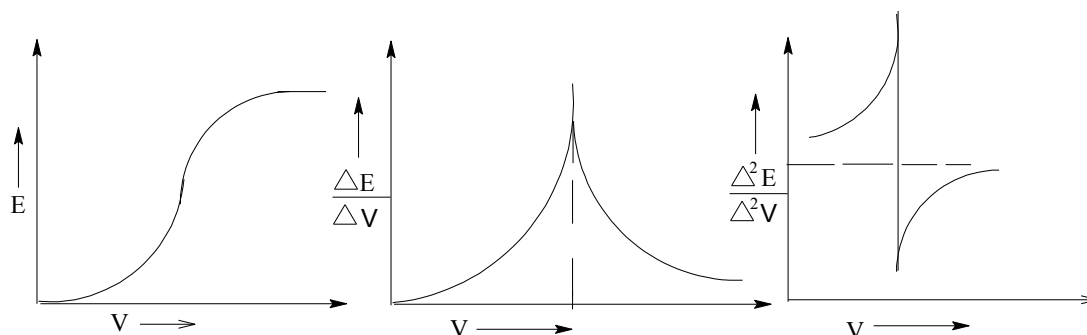
2. Red-ox titrations:

These are also carried out potentiometrically using an inert metal electrode like platinum. It is immersed in a solution containing both oxidized and reduced forms of same species.

$$\text{Oxidised form} + ne^- \longrightarrow \text{reduced form}$$

$$E = E^o + \frac{0.0591}{n} \log \frac{[\text{oxidised state}]}{[\text{reduced state}]}$$

The potential of the indicator electrode is controlled by ratio of oxidized and reduced forms. During oxidation or reduction, the ratio of concentration and hence the potential near the end point changes rapidly and can be readily detected. Thus titrations involving redox reactions can be followed potentiometrically, since at the equivalence point there will be a sudden change of potential. Platinum electrode is used as indicator electrode and saturated calomel electrode as reference electrode. Generally, oxidizing agent is taken in the burette.

3. Precipitation titrations:

Consider a titration of AgNO₃ Vs standard KCl, where Ag^+ ions are precipitated as AgCl. Using a silver electrode as indicator electrode & calomel as reference electrode, the potential can be measured as,

$$E = E^o + 0.0591 \ \log \ [Ag^+]$$

For the reaction, $Ag^+ + NO_3^- + K^+ + Cl^- \longrightarrow AgCl\downarrow + K^+ + NO_3^-$
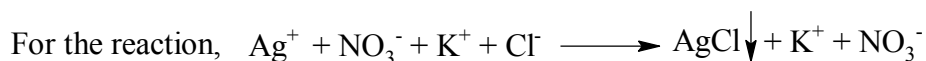
As KCl is added, Ag^+ ions are precipitated as AgCl. i.e., concentration of Ag^+ ions decreases during the process. The potential og Ag^+/ Ag goes on decreasing continuously on addition of KCl. At the end point, Ag^+ ion concentration is very small. Hence change in electrode potential is maximum. On continued addition of KCl, the concentration of Ag^+ ion remains almost same. Beyond the end point therefore, it causes a small change in electrode potential.

The potentiometric curves obtained are similar as obtained in acid base titration as described earlier.

CONDUCTOMETRY:

Principle:

According to Ohm's law, current (i) flowing in a conductor is directly proportional to emf (E) and inversely proportional to resistance (R).

Therefore, $i = \dfrac{E}{R}$

The reciprocal of resistance is conductance. The resistance of a homogeneous material of uniform cross section with an area of 'a' sq.cm and length 'l' cm is given by,

$R = \dfrac{\rho \ x \ l}{a}$, ρ is specific resistance

The reciprocal of specific resistance is specific conductance.

Specific conductance:

Specific conductance is the conductance of a solution present between 2 parallel electrodes of area 1cm² which are kept 1cm apart.

$K = \dfrac{1}{R} \ X \ \dfrac{l}{a}$ $\left[\dfrac{l}{a} = \text{cell constant} \right]$

The specific conductance of an electrolytic solution at any temperature depends on ions present and hence varies with ionic concentration. On dilution, specific conductance decreases as no of ions per ml also decrease.

Measurement of conductance is used to determine the equivalence point in acid base titrations. When indicators are used, the end point is detected by a change in colour. In conductometric titration, there is a sudden change in conductance of solution. Hence equivalence point is determined by plotting a graph of conductance versus volume. The principle behind the conductometric titration is replacement of ions of particular conductance by ions of different conductance. Eg: in titration of acid with a base, conductance of acid is due to H^+ ions, which are gradually replaced by Na^+ ions of base. Na^+ ions have lower mobility than H^+ ions & hence on continued addition of NaOH, conductance decreases, until it is completely neutralized. Further addition of base increases no of OH^- ions, & hence conductance increases. A plot of conductance versus volume of base gives 2 straight lines. The point of intersection of these gives the neutralization point.

ADVANTAGES:

✓ Mixture of acids can be titrated accurately.

✓ When visual or potentiometric methods fail due to solubility or hydrolysis, in such cases conductometric methods hold good.

✓ Accurate results are obtained in dilute as well as more concentrated solutions.

✓ The method can be employed for colored solutions also.

✓ Very weal acids which can't be titrated potentiometrically, can be titrated by conductometric method.

INSTRUMENTATION:

Conductometer consists of 2 platinum electrodes and a conductance measuring device. The 2 electrodes have unit area of cross section and are placed unit distance apart. The solution to be estimated is taken in a beaker. The system responds readily to changes in concentration of analyte.
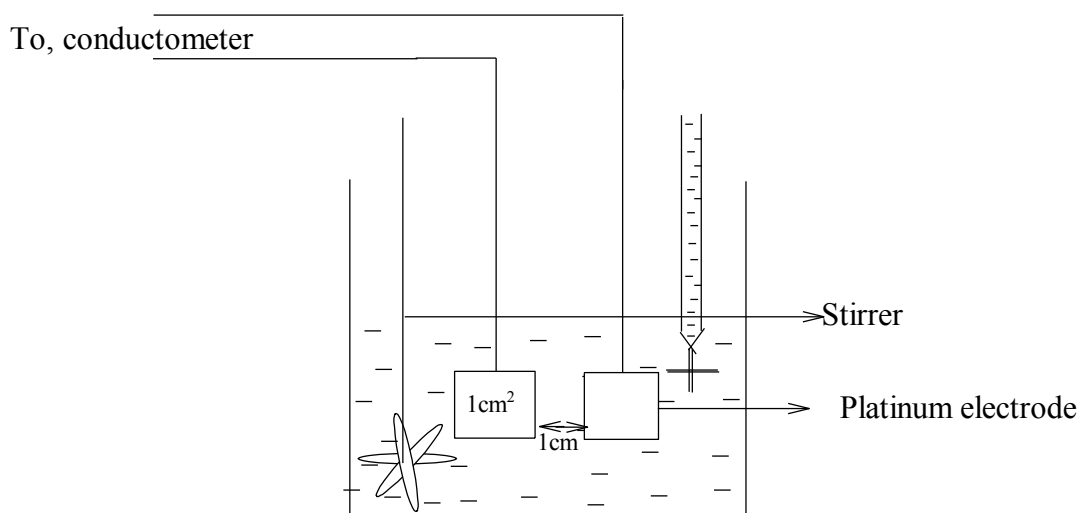
APPLICATIONS:

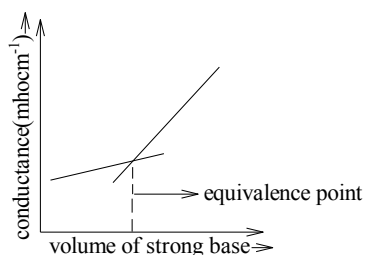**1. Titration of strong acid with a strong base:** eg: HCl Vs. NaOH

When strong acid is titrated with a strong base, conductance first falls since highly mobile H^+ ions are replaced by Na^+ of base. After the equivalence point, conductance rapidly increases with further addition of base due to increase in concentration of OH^- ions. The graph obtained on plotting conductance versus volume of base is as follows.
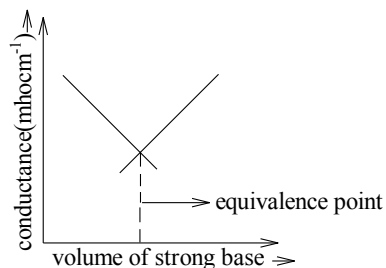

Two straight lines are obtained. The point of intersection is the end point.

2. Titration of weak acid with a strong base: eg: CH_3COOH Vs. NaOH

The conductance of weak acid is initially low, due to poor dissociation. When a strong base is added, the salt is formed, gets ionized and conductance increases slowly. On complete neutralization, further addition of base increases no of OH^- ions, and conductance increases sharply. The plot of conductance versus volume of strong base is as follows.

3. **Titration of acid mixture with strong base:**

In the mixture of strong acid and weak acid, strong acid dissociates completely and consumed first by the base. After the neutralization of strong acid, the weak acid is neutralized. During neutralization of the strong acid (Ex: HCl) conductance first falls since highly mobile H^+ ions are replaced by Na^+ of base. Later, during the neutralization of weak acid (Ex: acetic acid), the poorly conducting acid is converted into a salt (Ex. CH_3COONa). The salt undergoes complete dissociation, an therefore, the conductance increases gradually. After the complete neutralization, further addition of base NaOH introduces more and more Na^+ and OH^- ions into the solution, conductance again increases rapidly. When conductance of the solution is plotted against volume of base added, three straight lines will be obtained. First point of intersection corresponds to the volume of base required to neutralize strong acid and second point of intersection is for total acid.

11

equivalence point for strong acid
equivalence point
for total acid

Vol. of base ⟶
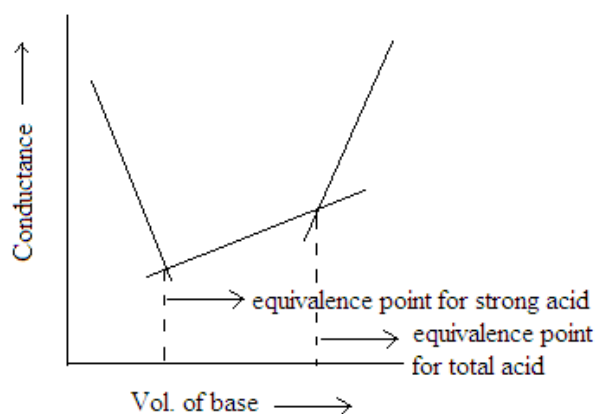
NANOMATERIALS:

Nanomaterials are the materials of which a single unit is sized between 1 and 1000 nanometers (10^{-9} meter). They have unique optical, electronic, or mechanical properties. The design, fabrication and application of nonmaterial is called nanotechnology. Nano particles can be naturally occurring or synthetically derived. Naturally occurring includes volcanic ash, soot from forest fires, wax crystals covering a lotus, butterfly wings, milk, blood and bone matrix. Synthetic nanoparticles include fullerenes, carbon nanotubes, metal or metal oxide nanoparticles. The fullerenes are a class of allotropes of carbon which conceptually are graphene sheets rolled into tubes or spheres. These include the carbon nanotubes (or silicon nanotubes) which are of interest because of their mechanical strength and electrical properties.

Nanomaterials are used in

- medicine for diagnostics, drug delivery, cancer therapy, synthetic bones, tissue engineering;
- In nanocosmetics for keeping the skin soft and wrinkle free;
- In textiles for inhibiting the growth of bacteria, fungi to avoid nail infections, foot odour and cracks in the heel;
- In nanosensors such as biosensors, chemical sensors, mechanical sensors, etc.
- In water purification
- In energy production, energy saving and in fuel cells
- In information and communication technology
- In heavy industry: construction, automotive, aerospace
- In consumer goods: food packaging, detection of contaminants in food, sporting goods, etc.

Properties of Nanomaterials (Size dependent):

The nanomaterials exhibit several size dependent properties, as follows.

Thermal properties:

The properties of nanoparticles change as their size approaches nanoscale. In bulk molecules the percentage of atom at the surface is relatively small to the total number of atoms of the material.

12

Due to small dimensions nanomaterials have extremely large surface area to volume ratio resulting in surface dependent material properties. Nanoparticles may have significantly low melting point due to huge fraction of surface atoms in the total amount of atoms. The number of bonds broken per atom during melting is less in nanomaterials than in macro particle, in which number of bonds need to be broken per atom is large due to large proportion of atoms in bulk.

Electrical properties:

In case of bulk molecules, there is delocalization of electrons and electron move in any directions. There is overlapping of orbitals in bulk particle. But in Nano scale there is discrete band gap. Hence metals which are good conductors in bulk, become semiconductor and finally insulator when size is decreased to Nano level.

Optical properties:

The discrete electronic states of nanoparticles allow absorption and emission of light of specific wavelength. Hence nanoparticles exhibit unique colors different from bulk. The optical properties of nanoparticles are determined by Surface Plasmon resonance phenomenon. When nanoparticles are much smaller than wavelength of light, coherent oscillation of conduction band electrons is induced by interaction with electromagnetic field. This resonance is called Plasmon resonance. This causes change in oscillation frequency of electrons, generating different optical properties including scattering and absorption.

Surface Area

If a bulk material is subdivided into individual nonmaterial, the total volume remains the same, but the collective surface area is greatly increased. For example surface area of a cube of $1m^3$ volume is $6\ m^2$. If this cube is cut into smaller and smaller cubes of $1nm^2$, we get 10^{27} cubes. The collective surface area of 10^{27} cubes is $6000Km^2$.

Therefore surface area increases largely on moving from bulk to nanoscale. In nanomaterials significant amount of atoms exist at the surface. The properties like catalytic activity, gas adsorption and chemical reactivity depend on the surface area. Therefore nanomaterials show specific surface related properties that are not observed in bulk materials.

Catalytic property

Because of the increase in the surface area of the nanomaterials, their chemical activity also increases. Due to increased chemical activity nanomaterials can be used as catalysts to react with toxic gases such as CO and NO in automobile catalytic converters and power generation units to prevent pollution from gasoline and coal. Bulk gold is catalytically inactive, but gold nanoparticles are catalytically very active for selective redox reactions. Noble metal nanoparticles are used as catalyst in fuel cells.

Synthesis of Nanomaterials:

The synthesis of nanomaterials is classified into two groups.

1. The top down method: where single crystals are etched in an aqueous solution for producing nanomaterials.

2. The bottom up method: where materials containing the desired precursors are mixed in a controlled fashion to form a colloidal solution.

Bottom up approach:

In bottom up approach the desired nanomaterial is built from the bottom, by orderly assembling species like atoms, ions or molecules. These are simple, economical methods which involve chemical technique and have better control over chemical composition, size and the products obtained are at high purity. Some of the methods used by this approach are discussed below.

1. **Sol-Gel process:**

It is a process used to prepare mono dispersed Nano particles of metal oxides at a low cost. A sol is colloidal suspension of solid particles dispersed in a liquid phase. A gel is colloidal solution made up of liquid particle dispersed in a solid phase. In general sol-gel consists of chemical transformation of sol to gel state and subsequent post treatment and transition to solid nanomaterials. Sol gel process can be explained by the following steps.

a. Preparation of sol:

In this process metal alkoxide (MOR), R an alkyl group is used as precursor (starting material) to synthesize nanoparticles. First sol is prepared by dispersing precursors in solvent (alcohol), and then hydrolysed with acidic or basic catalyst. Here alkoxide ligand is replaced by hydroxyl ligand.

$$MOR + H_2O \rightarrow MOH + ROH \text{ (hydrolysis)}$$

b. Conversion of a sol to gel:

Sol is further converted to gel by polycondensation reaction between MOH and MOR resulting in formation of oxide or alcohol bridged network (gel). These are complex multistep process involving gel formation.

$$MOH + ROM \rightarrow M\text{-}O\text{-}M + ROH \text{ (condensation)}$$

c. Aging of a gel (Syneresis): Gel on aging for a known period of time, condenses to nanoscale cluster of metal hydroxides.

d. Removal of Solvent: The solvent is removed from gel by evaporative drying. Here fundamental changes in the structure of gel are seen. A solid formed by dehydration of gel is xerogel. If the solvent is extracted under critical conditions, Aerogel is obtained. Aerogel is a synthetic porous ultralight material derived from a gel, in which the liquid component of the gel has been replaced with a gas.

e. Heat Treatment: The gel is heat treated at high temperatures to collapse the pores in the gel network and to drive out remaining organic contaminants.

Advantages of sol-gel process:

- High purity compounds are obtained

- The sol-gel approach is a cheap and low-temperature technique that allows for the fine control on the product's chemical composition.

- It is used to make ceramic and glass materials in the form of thin films, fibers, or powder.

2. Precipitation method:

In this method solid nano particles are obtained by careful precipitation from their solution. The process involves two steps: **Nucleation and Growth of particles**.

Precursors used in this method are hydrolysis products of metal salt (metal nitrate or acetate). Metal ions exist in the form of metal hydrates like $Al(H_2O)_6^{3+}$, $Fe(H_2O)_6^{3+}$. When a precipitating agent such as NaOH is added to the precursor solution, it changes the pH and causes condensation of precursors. Hence concentration of solution increases and reaches a critical level called super saturation. At this concentration, nucleus formation is initiated. The nucleus further grows into particles, which gets precipitated. Particle size of the product depends on rate of attainment of super saturation and rate of nucleation. If a solution attains super saturation level slowly and nucleation proceeds over a long period of time, then the precipitate with wide particle size is formed. On the other hand if a solution reaches super saturation and nucleation suddenly, then particles of smaller size are obtained. Thus, nano particles of desired size can be obtained.

The product obtained is filtered, washed with water, air dried and finally calcined at higher temperature to remove counter ions of metal salt like nitrate or acetate which are readily decomposed.

3. Chemical Vapour Condensation method:

It is a method that involves synthesizing nano particles from a gaseous phase by chemical reaction or decomposition of precursors at high temperature. The process is often used in the semiconductor industry to produce thin films. In CVC processes, precursors are vapourized and mixed with an inert carrier gas like N_2 and the mixture is fed into the reactor which is maintained at a required temperature. Precursors undergo reaction, producing a product which is deposited over the substrate surface to yield a coating of the required material. The byproducts and leftovers are passed on to the gas phase, and are easily removed. The reaction is catalysed by the catalyst present on the substrate. Most commonly used precursors are metal hydride, halides and organometallics.

Advantages:

- Simple technique and doesn't require ultra high vacuum.
- With CVC, it is possible to deposit nano materials with any shape and any size.
- The product obtained is highly pure.

Nano Scale Materials:

Nanoscale materials are a broadly defined set of substances that have at least one critical dimension less than 100 nanometers and possess unique optical, magnetic, or electrical properties.

Fullerenes:

Fullerenes are class of compounds made of only carbon atoms having closed cage like structures of hollow sphere, ellipsoid, tube, etc. Fullerenes with different number of carbon atoms like C_{60}, C_{70}, C_{74}, C_{76}, C_{78} etc have been prepared and investigated. C_{60} molecule has shape like a soccer ball and is called Buckminster fullerene.

Structure: Fullerenes have cage like structure built up of fused pentagons and hexagons. The pentagons provide curvature required to form closed structure. All fullerenes contain 12 pentagons and different number of hexagons. Each fullerene has $2(10+M)$ number of carbon atoms corresponding to 12 pentagons and M number of hexagons. C_{60} molecule has 12 pentagons, 20 hexagons, 90 edges and 60 vertices. All carbon atoms are sp^2 hybridized and trigonally bonded, similar to graphite.

Synthesis:

Fullerenes are prepared by vaporization of graphite at very high temperature and condensing the vapors in inert atmosphere. Graphite is vaporized by setting up an electric arc between two graphite electrodes in a controlled atmosphere of helium gas. The temperature at the tip of the electrode is more than 4000°C and pressure of He gas is 150-200 torr. A mixture of various fullerenes is obtained, out of which the main product is fullerene C_{60}. It is extracted and crystallized using benzene solvent.
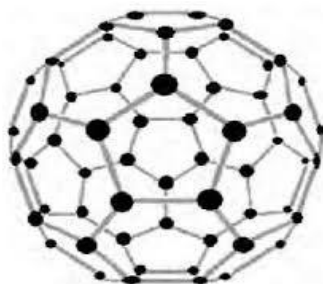
Uses:

It is used as an antioxidant to trap free radicals generated during an allergic reaction

It is used to store hydrogen, possibly as a fuel tank for fuel cell powered cars.

In electronic industry, it can be used in preparation of modular switches, diodes, transistors.

It is used in optical fibers because of the perfect spherical shape and their ability to transmit light.

Carbon Nano tubes:

Carbon nanotubes (CNTs) are allotropes of carbon with a cylindrical nanostructure. Nanotubes are members of the fullerene structural family. Their name is derived from their long, hollow structure with the walls formed by one-atom-thick sheets of carbon, called graphene. These sheets are rolled at specific and discrete angles, and the combination of the rolling angle and radius decides the nanotube properties.

Nanotubes are categorized as single-walled nanotubes (SWNTs) and multi-walled nanotubes (MWNTs). Individual nanotubes naturally align themselves into "ropes" held together by van der waals forces. The diameter of tubes is typically of nanometer dimensions, while lengths are in micrometers range.

Synthesis: CNTs have been prepared by chemical vapor deposition (CVD) process from hydrocarbons using iron or cobalt catalysts. Propylene is fed into the reactor maintained at 800°C with carrier N_2 gas. It undergoes pyrolytic decomposition depositing uniform layer of carbon on the inner wall of the template (anodic aluminium oxide, AAO) nanochannels. The AAO template is removed by washing with NaOH, only CNT is left as an insoluble product. The diameter and thickness of the resulting nanotube is controlled by changing the inner diameter and length of the nanochannel.
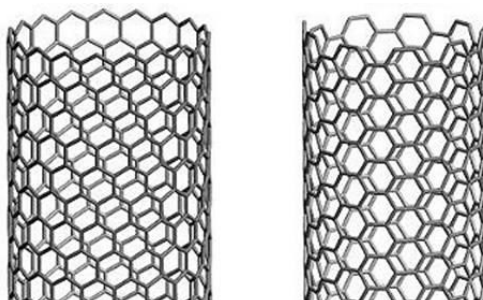
Properties: CNT's have hundred times the tensile strength of steel, high electrical and thermal conductivity, high mechanical strength with high thermal resistivity.

Uses:

CNTs can serve as a multifunctional coating material.

Single-walled nanotubes (SWNTs) have potential for use in solar panels, due to their strong ultraviolet absorption characteristics.

CNT can be used for desalination and in Biomedical Applications.

Carbon nanotubes can be used as filters for cleaning polluted water.

Graphene:

Graphene is a one-atom-thick layer of carbon atoms arranged in a hexagonal lattice. It is the building-block of Graphite (which is used, among others things, in pencil tips), but graphene is a remarkable substance on its own - with a multitude of astonishing properties which repeatedly earn it the title "wonder material" .Graphene is a crystalline allotrope of carbon with 2-dimensional properties. Each atom has four bonds: one σ bond with each of its three neighbors and one π-bond that is oriented out of plane. The atoms are about 1.42 Å apart. All carbon atoms are sp^2 bonded to adjacent carbon atom.

Applications: Grapheme is a flexible transparent conductor because of it is used in solar cells, LEDs, touch panels, Field effect transistor.

Dr. Priya V Frank
Associate Professor
Dept. of Chemistry
Canara Engineering College.