Module - 6
NANO CHEMISTRY

- Nanochemistry deals with the study of synthesis, characterization and applications of nanomaterials.

Q.1 What is nanomaterials? Give some examples.

Ans. Nanomaterials are those substances whole length or diameter or thickness lies between 1 - 100 nm.

Here, $1\ nm = 10^{-9}\ m$.

Examples:- Fullerene (diameter is ~ 1 nm)
Gold nanoparticles ~ 50 nm

- In general, nanomaterials exhibits improved physical, chemical, optical,

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medicinal, thermal catalytic properties than bulk materials.

for example,

(1) A drop of water (d ~ 5 nm) boils at 95.9 °C compared to a large quantity of water.

(2) Antibacterial properties increases in the nano-range

(3) Adhesive property increases in the nano-range

(4) Catalytic activity increases by decreasing size

(5) Thermal property improves on decreasing size

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Q.2 Give reason that nanomaterials shows better properties than bulk materials.

Ans:- It is due to the large surface area to volume ratio.

For a spherical particle

$$\frac{\text{Surface Area}}{\text{Volume}} = \frac{4\pi r^2}{\frac{4}{3}\pi r^3} = \frac{3}{r}$$

spherical particle

r = radius of particle

$\Rightarrow$ the ratio $\uparrow$ as r $\downarrow$ i.e size decreases.

Synthesis of Nanomaterials

In general, nanomaterials (NMs) are synthesised by two approaches:

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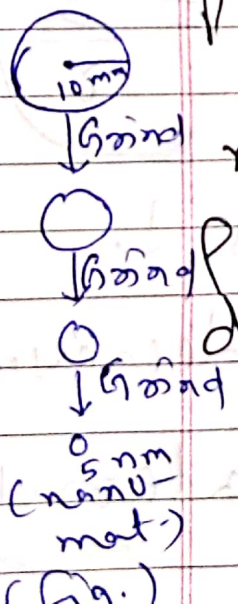
1. Top-down approach
and
2. Bottom-up approach

Q.3 What is Top-down method? Give an example.

Ans) It is a solid state process in which large particles (in the range of mm or μm) are converted into nanomaterials by mechanical processes like crushing, grinding, Ball milling, etc.



(Fig.)

Example:- High energy ball milling

Q.4 Mention some limitations of Top-down approach.

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Ans:- (i) Not Suitable for large scale production

(ii) time consuming process

(iii) Synthesized NMs contains defects

(iv) NMs obtained are not of uniform size

Q.5 What is Bottom-up approach? mention some of its advantages.

Ans:- It is a process by which NMs are obtained by atom-by-atom or molecule-by-molecule or cluster-by-cluster combination.

Examples:- Sol-gel process chemical reduction, combustion, chemical vapour deposition, microwave

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Synthesis, etc.

Advantages :-

(i) Relatively a fast process
(ii) Develops particles of uniform size & shape
(iii) Develops particles of excellent quality & of good quantity
(iv) Environmental friendly process
(v) Usually NPs are free from any imperfections

Green Synthesis of NPs

Q.6 What is green synthesis? Give an example.

Ans: Green synthesis is a

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bottom-up approach where non-toxic safe reagents which are eco-friendly are used for the synthesis of NMs.

Example:- Eco-friendly reagents are plant extracts i.e extracts of leaves, fruits, barks, flowers, etc.

Q.7 Mention some advantages of green synthesis.

Ans) (i) Minimises generation of waste

(ii) uses non-toxic reagents

(iii) fast process

(iv) produces NMs of uniform size, shapes & distributions

(v) low cost process

(vi) Plant extract contains both reducing agent & capping agent.

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Synthesis of nanoparticles (NPs)

Q.8 Mention the raw materials needed to develop NPs.

Ans Three important raw materials needed are:

(i) Precursor salt:- It is a salt from which NPs can be obtained.
For example,

Precursor salts
- $AgNO_3$ → gives Ag-NPs
- $AuCl_3$ → " Au-NPs
- $Zn(NO_3)_2$ → " ZnO-NPs
- $CuSO_4$ → " Cu_2O-NPs

(ii) Reducing Agent:- It reduces the metallic cations of precursor salt to Atomic

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form.

for example,

$$Ag^{+} + e \xrightarrow{Red^{n}} \underset{(Ag\text{-atom})}{\overset{\text{ox-state}}{Ag^{(0)}}}$$

many Ag-atoms combine to form Ag-NPs

examples:- Citric Acid, Hydrazine (N_2H_4), Plant extract (fr. of Phenols, Alkaloids, etc.)

(iii) Stabilizing / Capping agent:-

It stops agglomeration and growth of NPs.

examples:- Poly vinyl Alcohol, Surfactants, Plant extracts, etc.

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Q.9 What is a Capping agent? Give an example.

Ans:- A Capping agent is a substance which when added to the precursor salt solution checks agglomeration of NPs.

example:- ~~Citric Acid~~ Poly vinyl Alcohol

Q.10 Name a precursor salt used to synthesize Silver nanoparticles.

Ans:- $AgNO_3$ (Silver Nitrate)

Q.11 Give an example of a reducing agent used in synthesis of NMs.

Ans:- Citric Acid

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÷ Long Questions ÷

Q.1) Discuss the Synthesis characterisations and applications of Gold nanoparticles (NPs).

Answer

① Synthesis of Gold (Au) NPs

(i) Preparation of Plant extract:

Take 10 g of Papaya leaves and cut into small pieces. The cut pieces are taken in a 200 ml beaker containing 100 ml of distilled water. Place the beaker on a hot magnetic stirrer and boil it for 30 minutes. Then cool it and filter it to get the aqueous extract solution.

(ii) Preparation of Precursor salt solution:

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Take 10 mg of gold salt $HAuCl_4$ and dissolve it in 10 ml of water to get the aqueous solution.

(iii) Preparation of Au-NPs :-

Take 10 ml of aqueous plant extract solution in a 50 ml beaker and place it on a magnetic stirrer maintained at 50 °C. Add 1 ml of gold solution into dropwise using a micro-pipette. The solution turned pink indicating development of Au-NPs.

$$HAuCl_4 (Au^{+3}) + 3e^- \longrightarrow Au$$

(2) Characterisation of NPs :

The developed Au-NPs

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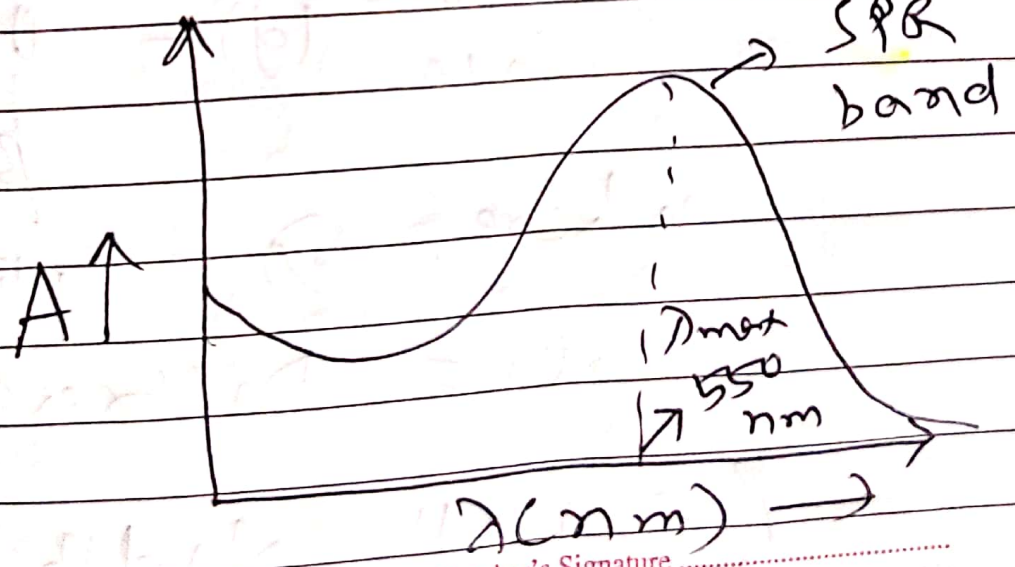
were characterised using UV-Visible spectrophotometer and x-ray diffractometer.

(i) UV-Visible Analysis :-

A broad absorption band at 550 nm shows formation of Au-NPs. This band is known as surface plasmon resonance band (SPR-band).

SPR-band results from the resonant oscillations of free electrons of metal atoms.



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(ii) X-ray diffraction (XRD) Study

XRD study helps us in knowing the crystalline nature, crystal type and crystallite size of Au-NPs.

In the XRD-pattern, a peak at $2\theta = 38.16^0$ corresponds to (111) plane shows formation of FCC Au-NPs.

Crystallite size can be calculated using the Debye-Scherrer equation:

$$\text{Crystallite size } (d) = \frac{0.94\lambda}{\beta \cos\theta}$$

where, $\lambda = 1.54\ A^0$

θ = Diffraction angle in radians

β = full width at half maximum (FWHM)

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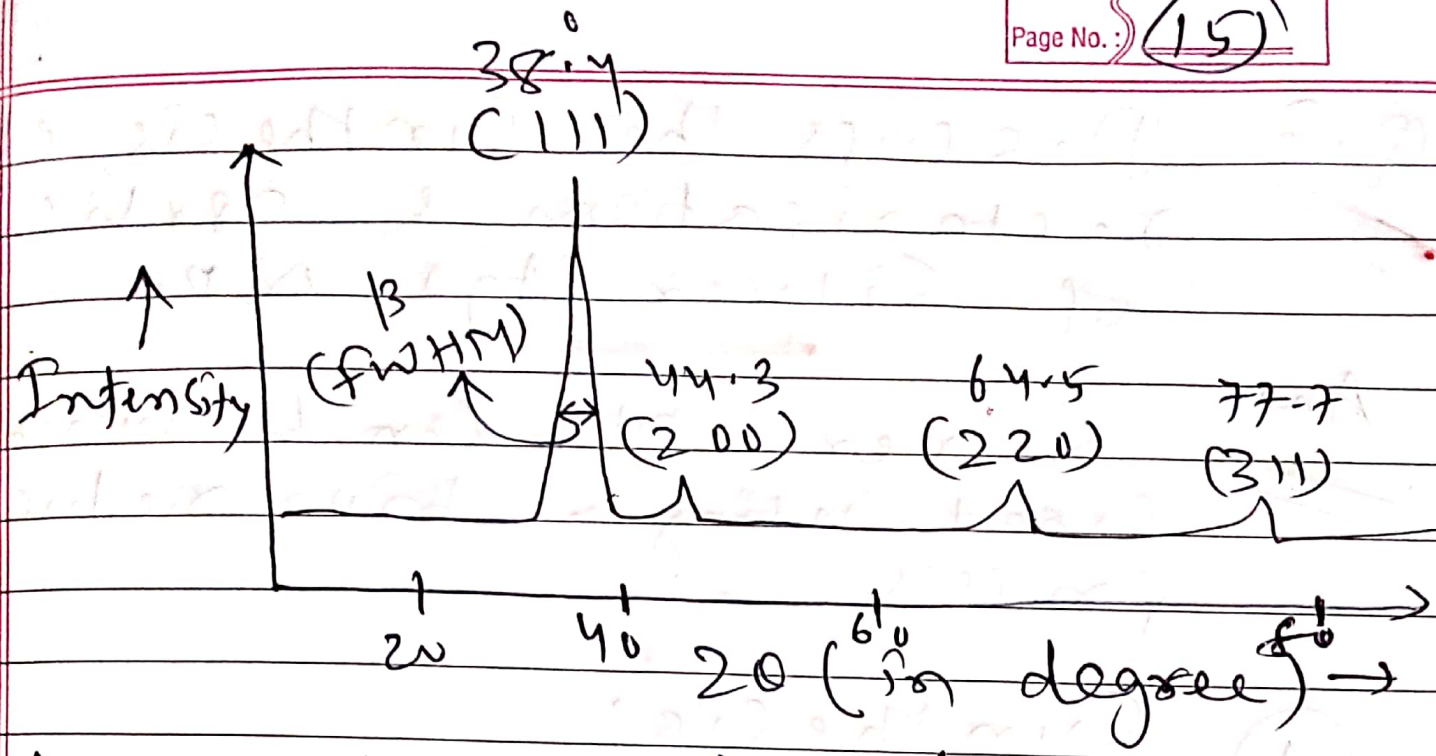

Applications of Au-NPs:

1. In drug delivery system
2. As a catalyst
3. As bio-sensor
4. Antibacterial & anti-fungal agent
5. In photodynamic Therapy
6. In cosmetics
7. In water treatment, etc.

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Q.2 Discuss the synthesis, characterisations & applications of Silver (Ag) NPs.

Ans. Silver NPs can be synthesised using a bio-reduction process.

(A) Synthesis:

(i) Preparation of plant extract

Take 10 g of neem leaves in a 200 ml beaker. Add 100 mL of distilled water to it and placed on a hot magnetic stirrer maintained at 100°C for 30 min. Cool the beaker and filter it to get clear aqueous plant extract.

(ii) Preparation of $AgNO_3$ soln:-

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Take 10 mg of $AgNO_3$ salt and dissolve it in 5 ml of distilled water.

(iii) Development of Ag NPs :-

Take 10 ml of extract soln in a 50 mL beaker. placed it on a hot magnetic stirrer maintained at 40°C. Under hot stirring condition add 1 ml of $AgNO_3$ soln to the beaker. The colour suddenly changed to yellowish brown indicating formation of Ag-NPs.

$$Ag^+ (AgNO_3) + e^- \xrightarrow{Red^n} Ag$$

(B) Characterizations :- The sample was characterised using UV-Vis spectrophotometer and X-ray diffractometer.

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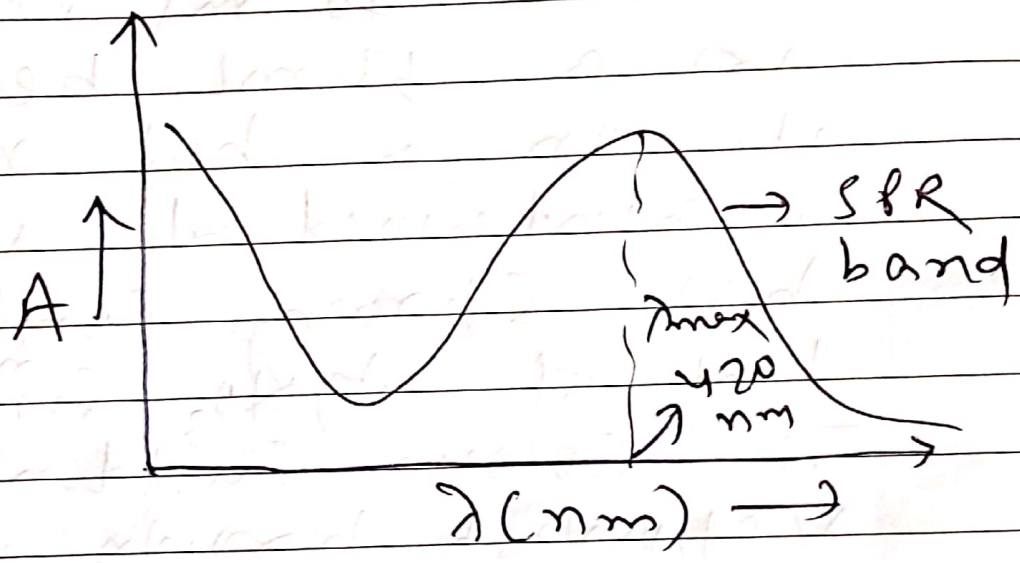
(i) UV-Visible Study:

In the absorption spectrum a broad band near 420 nm known as SPR-band shows formation of Ag-NPs.



(ii) XRD Study:-

XRD study helps us in knowing:

(a) Lattice type

(b) Crystallite size, and

(c) Crystalline nature of Ag-NPs

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In the XRD-pattern an intense peak at 38° corresponds to (111) plane of fcc-type Ag-NPs. The crystallite size can be find using Debye-Scherrer eqn -

$$d = \frac{0.94\lambda}{\beta \cos\theta}$$

(c) Applications :- Some applications are :

1. In cosmetics
2. As antibacterial agent
3. In water treatment
4. In food packaging
5. As bio-sensors
6. As catalyst
7. In coatings
8. In textile industries

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Q.3 Discuss the synthesis characterisations and applications of Cuprous oxide (Cu_2O) NPs.

Ans:- (A) Synthesis of NPs:-

To synthesize Cu_2O NPs, first prepare the plant extract, then prepare the salt soln and mix the two solution under hot condition.

i) Preparation of Plant extract:- Take 20g of Arka leaves. Cut into pieces and take in a 500 ml beaker. Add 200 ml of water and boil it for 30 min. Cool the beaker and filter it.

(ii) Preparation of Precursor salt solution:- prepare 1M solution of Copper Sulphate by dissolving ~~in~~ suitable

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amount of $CuSO_4.5H_2O$ blue crystals in required volume of water.

(iii) Development of Cu_2O NPs :-

Take 10 ml of plant extract in a 50 ml beaker and then placed on a magnetic stirrer maintained at 70°C. Then add 2 ml of $CuSO_4$ soln to it in a drop wise manner. Add few drops of hydrazine soln to it. till the colour changed to reddish brown suspension from blue. The reddish brown particles are Cu_2O NPs.

(B) Characterisations of NPs :-

The Cu_2O NPs were characterised by UV-VIS Spectrophotometer and X-ray diffractometer.

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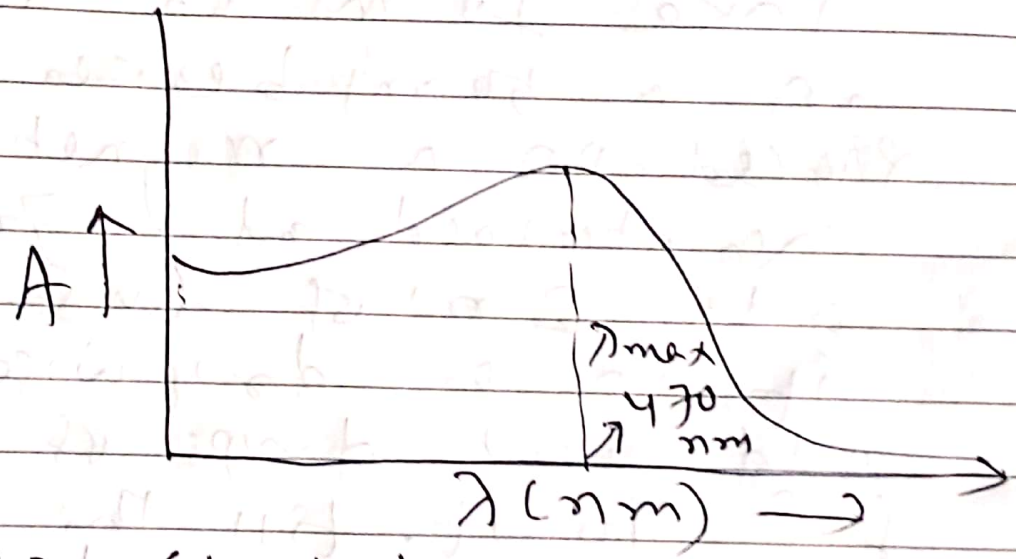
(i) UV-Visible Study:-

On the absorption spectrum a broad band (SPR band) found near 470 nm suggests formation of Cu_2O - NPs.



(ii) XRD Study:-

Characteristics XRD peaks found at 36.8° and 42.3° corresponds to (111) and (200) crystal plane suggests formation of Cu_2O-NPs.

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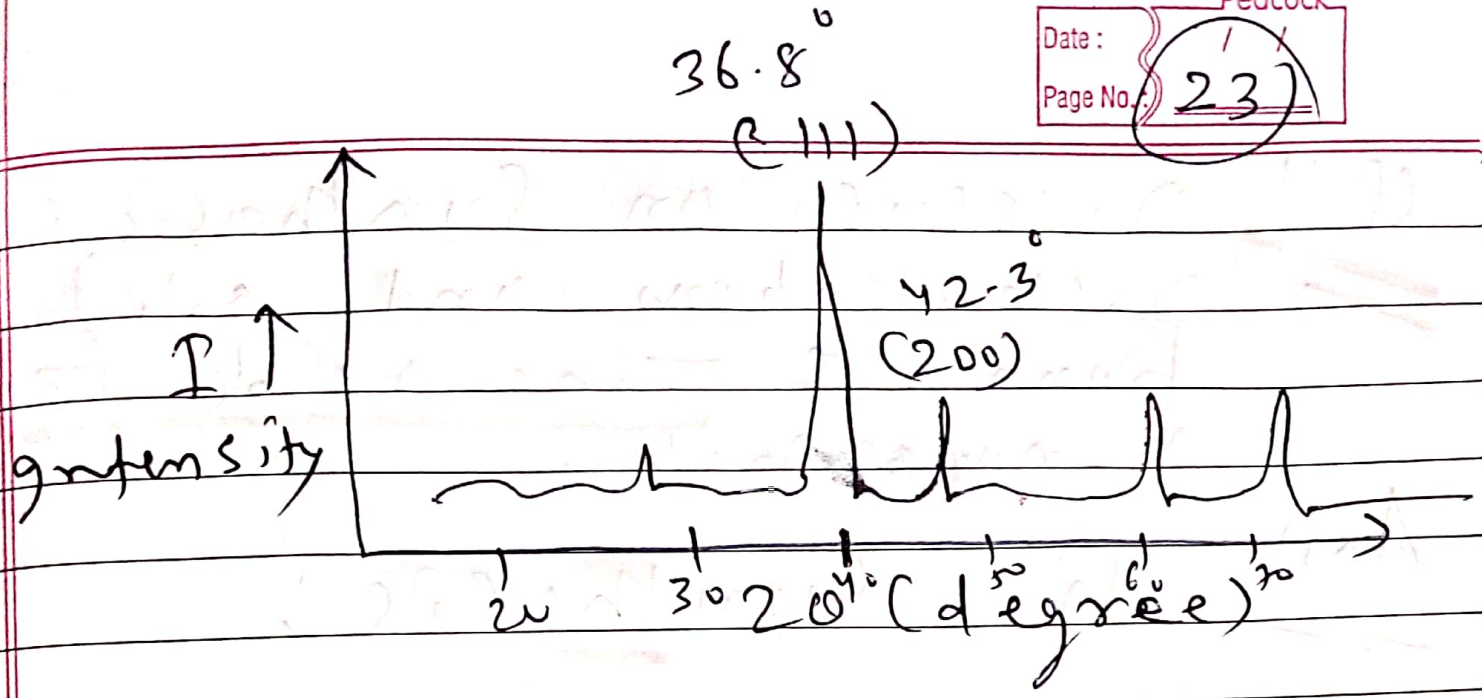

Formation Mechanism :-

$CuSO_4 - 5H_2O \xrightarrow{Hyd.} Cu(OH)_2$

$Cu(OH)_2 \xrightarrow{Heat} Cu_2O$ - NPs

(C) Applications :-

1. As Photo-Catalyst
2. In Water Treatment
3. As Anti-bacterial Agent
4. In Solar cells
5. In magnetic Storage devices

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Q.4 Discuss on Synthesis, characterisations and applications of Zinc Oxide (ZnO) nanoparticles.

Ans:- (A) Synthesis:

(i) Preparation of Plant extract:- 20 gm of Hibiscus leaves was boiled in a beaker containing 200 ml of water for 30 minutes. Then cooled the beaker and filter it to get the extract.

(ii) Preparation of Salt Solution:-

Take 10 mg of $Zn(NO_3)_2$ salt and dissolve it in 20 ml of water to prepare salt soln.

(iii) Development of ZnO-NPs:-

Take 20 ml of extract

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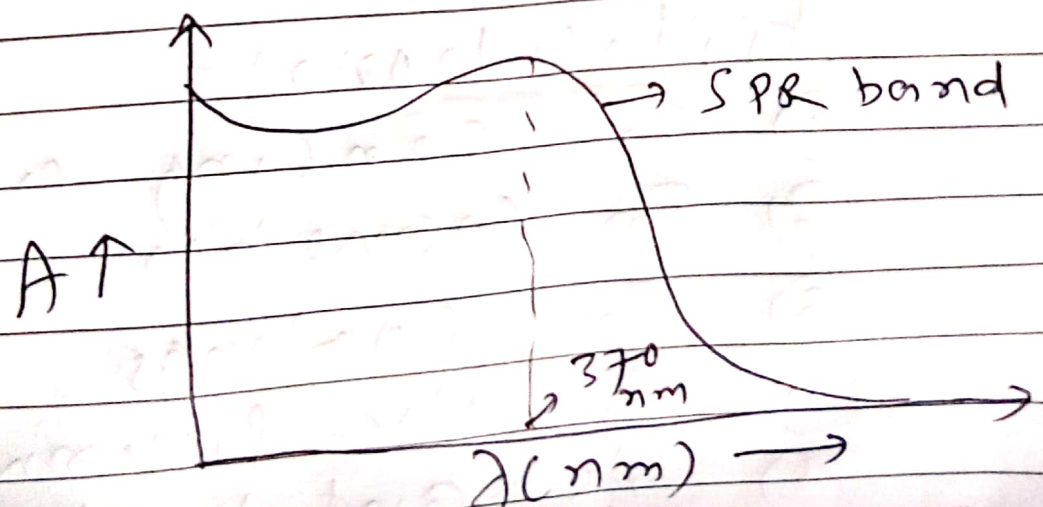
in a 100 ml beaker and placed on a hot magnetic stirrer maintained at 90°C. Then add 2 ml of $Zn(NO_3)_2$ soln to the hot soln. Now add 1 ml of NaOH soln to it. White precipitates of ZnO was collected at the bottom of the beaker. Filter it and collect the powder.

(B) Characterisations:

(i) UV-visible study:-

A broad SPR-band at 370 nm shows the formation of ZnO-NPs.



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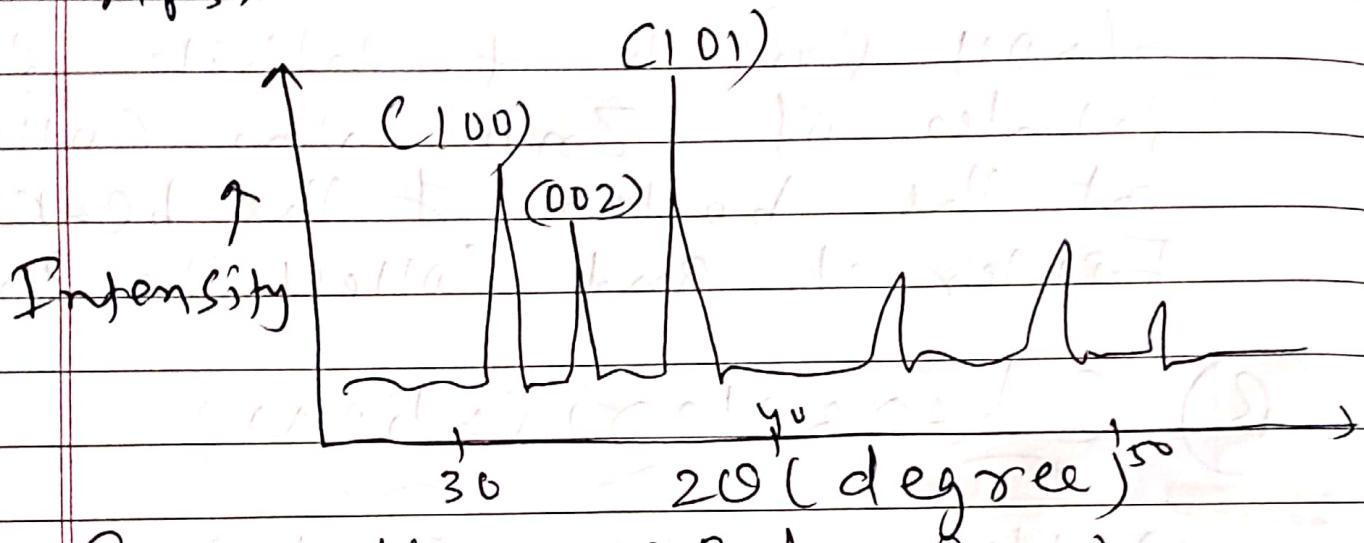
(ii) XRD - Study :- XRD - Pattern shows 2θ - values at 31.77°, 34.4° and 36.2° corresponds to (100), (002) and (101) planes of ZnO-nps.



Formation mechanism :-

$$Zn(NO_3)_2 \xrightarrow{Hyd.} Zn(OH)_2$$

$$Zn(OH)_2 \xrightarrow{\Delta} ZnO \text{ (NPs)}$$

Applications :-

1. In sensing applications
2. In cosmetics
3. In coatings
4. As UV - filtering Agent
5. As anti-bacterial agent

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