**Synthesis and Characterization of MoO3 Nanorods**

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**Abstract:-**

In the present investigation we have synthesised MoO3 nanoparticles by chemical bath deposition. The synthesised MoO3 nanoparticles are well indexed to hexagonal crystal structure. The morphological study reveals presence of nanorods with hexagonal crystal structure. The optical study shows direct allowed band gap with absorption in ultra violet region. The compositional analysis shows presence of molybdenum and oxygen in the synthesised nanoparticles.

**Keywords** :- MoO3, CBD, nanorods

**Introduction:-**

Recently, the transition metal oxides (TMOs) attracting a great deal of attention in the field of material science due to its variety of crystal phases and properties. The nano crystalline transition metal oxides such as TiO2, MoO3 and ZnO are most widely studied by various researchers all over the world. Among the transition metal oxides Molybdenum trioxide (MoO3) exhibit better intercalation chemistry with unique chemical, electrochemical, electronic and catalytic properties.

MoO3 is the most produced product of molybdenum worldwide than any other molybdenum compound due to the comparative instability of molybdenum oxides of lower oxidation states.

The MoO3 exhibits variety of crystal phases such as orthorhombic (α-MoO3) and hexagonal (h-MoO3). From these two hexagonal form of MoO3 is metastable which orthorhombic form of MoO3 is thermodynamically more stable than any other form.

Molybdenum trioxide (MoO3) exhibits excellent structural, chemical, electrical, catalytic, and optical properties . They possess a perovskite-like structure which makes them a suitable candidate for optoelectronic applications. Several studies were carried out to know more about the optical, structural and morphological properties of MoO3. Potential applications of MoO3 include sensors, catalysts, fuel cells, solar cells, supercapacitors, memory devices, etc.

**Experimental:-**

Chemical bath deposition method is used for the synthesis of MoO3 nanomaterials. In a typical synthesis ammonium heptamolybdate tetrahydrate (AHM) and conc. Nitric acid (HNO3) is used as a precursor for the reaction.

In a experimental setup, 15ml of 0.05M AHM solution was taken in reaction bath. The temperature of solution was increased slowly up to 500 C by using heating mantle. To this 5ml of concentrated HNO3 was added in a drop wise manner with constant stirring to obtain a clear solution. This clear solution was then stirred for 15 minutes and temperature of the reaction bath was maintained to 700 C for 30 minutes to obtain the yellowish white coloured precipitate of h –MoO3. Then the reaction mixture was cool at room temperature. Finally, the precipitate of h-MoO3 was filtered by Buchner funnel using whatman filter paper no 42. Then the precipitate was washed with hot distilled water and product was dried in muffle furnace at 500 C for 2 hours.

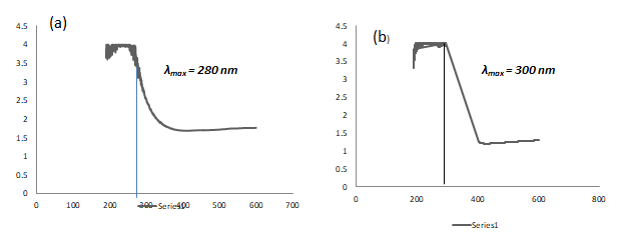
**Growth and reaction mechanism**:-

The growth of nanoparticles by CBD follows Ostwald ripening law. According to the Ostwald ripening law, the number of smaller crystallites sacrifices themselves to form larger crystallites. As a result of this initially the seed nuclei is formed by using the Ostwald ripening. After this these seed nuclei combine to form a multi-nucleation centres. The uniform growth of the nanoparticles is possible due to ripening of multi-nucleation centres. Hence to obtain a desired morphology control over the process of nucleation or growth is the most important aspect. Hence by controlling the parameters of CBD we have obtained the growth of nano rods for the MoO3. The nano rods are 1D in nature with hexagonal cross section. The 1D nanomaterial is having a higher surface area. Hence they show a better catalytic activity. The reactions which are taking place are as follows,



**Optical study :**

In order to study optical properties of MoO3, optical absorption of h-MoO3 and α-MoO3 is recorded on UV spectrophotometer in range 190 to 700nm, the absorption spectra clearly indicates that MoO3 is active in UV region. The band gap for MoO3 samples are calculated by the classical absorption equation. It is found that the λmax value for h-MoO3 is280nm and for α-MoO3 λmax is 300nm. From λmax values of both the samples it is clear that the band gap for MoO3 is near about 3eV. From this we can conclude that MoO3 is wide band gap semiconductor material.



***Fig 1 : (a) Graph of h- MoO3 ( Wavelength vs Absorbance) (b) Graph of α MoO3 ( Wavelength vs Absorbance).***

**Structural study :**

The structural study aspects of MoO3 nanoparticles was determined by using X-Ray diffraction. XRD pattern of the samples h-MoO3 is shown in figure 2,



Fig 2. XRD pattern of h-MoO3

The crystal structure and phase identification of MoO3 sample was carried out by using X-ray diffraction analysis. Here we have synthesized h-MoO3 nanoparticles. All the peaks in the XRD are well indexed to the JCPDS card no. 21-0569. No extra peaks due to impurity are seen.

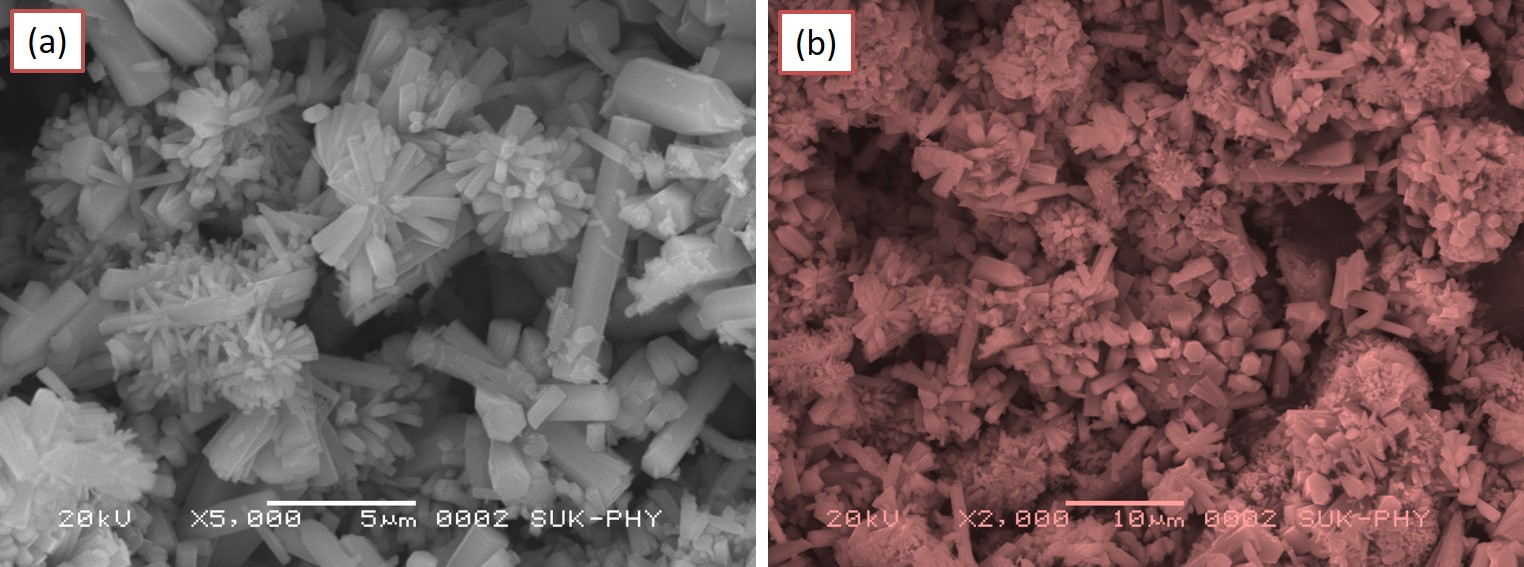
The crystallite size is calculated by using equation,

D= 0.9λ/βCosƟ

The crystallite size is calculated by using most intense (210) peak. The crystallite size of h-MoO3 is 35nm.

**Morphological study :-**

Morphology of nanomaterials plays a crucial role in physical and chemical properties of nanomaterials. To study the morphological aspects of MoO3 nanopaticles. The surface morphology of both h-MoO3 was characterize by SEM analysis. Figure below clearly indicates presence of 1D hexagonal rods. All the nanorods are assembled together to form nanoflower like structure. The size of each nanoflower is about 1 micron.



**Fig.3** SEM micrographs of MoO3 nanorods

**Compositional analysis:-**

In order to confirm the composition of a nanomaterial EDS analysis is carried out. The presence of Mo and O in synthesised nanopowder is shown in the EDS spectra.

The observed and actual atomic percentage is in good agreement**.**

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**EDS spectrum of MoO3 thin film**

**Conclusions:-**

The MoO3 nanopowder is synthesized by chemical bath deposition. The structural study shows hexagonal crystal structure. The SEM micrographs reveals nanorod like structure. The EDS spectra confirms presence of molybdenum and oxygen. The optical study reveals the absorption in the Ultra violet region. All these properties reveals that MoO3 is a better candidate for gas sensing .

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