**Synthesis and Characterization of MoO3 Nanorods**

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**Abstract:-**

In this research, we synthesized MoO3 nanoparticles by chemical bath deposition. The synthesized MoO3 nanoparticles are well indexed to the hexagonal crystal structure. A morphological study reveals the presence of nanorods with a hexagonal crystal structure. An optical study shows a direct allowed band gap with absorption in the ultraviolet region. Compositional analysis shows the presence of molybdenum and oxygen in the synthesized nanoparticles.

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

**Introduction:-**

Recently, transition metal oxides (TMOs) have attracted much attention in the field of materials science due to their variety of crystalline phases and properties. Nanocrystalline transition metal oxides such as TiO2, MoO3 and ZnO are the most studied by various researchers worldwide. Among transition metal oxides, molybdenum oxide (MoO3) exhibits superior 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.

MoO3 exhibits different crystal phases such as orthorhombic (α-MoO3) and hexagonal (h-MoO3). Of the two hexagonal forms, MoO3 is metastable, with the orthorhombic form of MoO3 being more thermodynamically stable than any other form.

Molybdenum oxide (MoO3) exhibits excellent structural, chemical, electrical, catalytic and optical properties. They have a perovskite-like structure, which makes them a suitable candidate for optoelectronic applications. Several studies have been carried out to learn 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 the experimental setup, 15 mL of 0.05 M AHM solution was placed in the reaction bath. The temperature of the solution was slowly increased up to 500 C using a heating jacket. To this, 5 mL of concentrated HNO 3 was added dropwise with constant stirring to give a clear solution. This clear solution was then stirred for 15 min and the temperature of the reaction bath was maintained at 700 C for 30 min to obtain a yellowish-white precipitate of h –MoO3. The reaction mixture is then cooled to room temperature. Finally, the precipitate of h-MoO 3 was filtered off with a Buchner funnel using Whatman No. 42 filter paper. Then, the precipitate was washed with hot distilled water and the product was dried in a 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:-**

The morphology of nanomaterials plays a fundamental role in the physical and chemical properties of nanomaterials. To study the morphological aspects of MoO3 nanoparticles. The surface morphology of both h-MoO3 was characterized by SEM analysis. The image below clearly shows the presence of 1D hexagonal rods. All the nanorods are assembled together to form a 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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**Reference id - IIPER1655365759**