Synthesis and characterization of Molybdenum Trioxide nanobelts by Hydrothermal and Chemical method: A comparative study

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Abstract. An ntype semiconductor with wide gap, Molybdenum trioxide provides as an apt material with its layered crystal structure. It has many applications such as hydrogen evolution, field emission thin film transistor, supercapacitors, gas sensors and energy emission devices. In the present work molybdenum trioxide nanobelts are synthesized by hydrothermal method and chemical method and a comparative study is made. The XRD SEM and EDS are done for the samples prepared and the results are analysed. XRD pattern shows sharp peaks indicating that the as synthesized molybdenum trioxide is crystalline in nature. The EDS pattern shows consistency in the weight percentage of elements. The crystal grain sizes were determined in both the cases.

Keywords: Hydrothermal method, chemical method, XRD, SEM, EDS.

1. Introduction

With the advancement of micro and nano technologies, it is possible to alter the structure and properties of materials. The quality of our lives has improved by the evolution of nano technology [1]. The applications in nano technology led to its processing and manufacturing. Nanotechnology covers Engineering Science that integrates Chemistry, Physics, Electronics, Material Science, and also Biological Sciences. The area of nanotechnology deals with the development of nanomaterials by Physical, Chemical processes, assembling of nanomaterials, and fabrication of nano devices, opto, electro-mechanical systems. Chemical synthesis, spontaneous self- assembly of molecular cluster, are methods by which nano materials are built up. The opportunities for developing innovative nano systems and nano materials are done with the discovery of novel materials, and phenomena and the development of new experimental and theoretical processes [5,6].

Literature survey: Literature review is an important part of scientific research study. It helps the researcher to identify a novel problem, helps to bridge the gaps in the research area and also empowers the scholar with new insight into the problem of study.

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New metal oxide nano-particles (MONPs) which are developed possess remarkable properties at low cost which gives interesting results for umpteen applications. With the development of nano- technology the various applications of MONPs consists in energy conversion, optical imaging, data storage, antenna etc.

Molybdenum oxide has been excellently focussed due to its high theoretical capacity, because of its high voltage and energy storage. Molybdenum trioxide nanoparticles exhibited good electro chemical sensing property to detect toxic material lead. With itsspecific layered structure and unique electrochemical property molybdenum trioxide happens to be a fascinating transition metal oxide [9,10].

There are variety of applications for molybdenum oxide NP's. Some of them are as sensors and electrode materials with high theoretical capacity. They can be used in chemical synthesis, in petroleum refining, and in recording media Molybdenum trioxide is photosensitive that changes colour when light falls on it.

Molybdenum trioxide materials havea higher theoretical capacity when compared to graphene, which is usually used as electrodes for anodes in lithium-ion batteries.

In the field of energy storage, they find application as cathodes or anodes for ion batteries like (sodium-ion batteries, potassium-ion batteries, etc.), and electrodes for supercapacitors.

Molybdenum trioxide finds wellrecognized applications in electronics, catalysis, sensors, field emission devices, lubricants, superconductors, thermal materials, biosystems, chromogenics and electrochromic systems [7,8,10,11].

The MoO_3 crystallises in three polymorphs orthorhombic MoO_3, Monoclinic MoO_3 and hexagonal MoO_3. Being thermodynamically stable and with layer structure the Orthorhombic phase is of greater significance in the scientific and Industrial fields. Different morphologies of orthorhombic MoO_3 like nanowires, nanotubes nanoplates, nanorods and nanobelts have been reported so far. Due to its single crystalline nature belt -like orthorhombic MoO_3 has attracted attention, which has special characteristics over bulk crystals [4,12].

2. Methods and materials

2.1 Hydrothermal method

Hydrothermal method is feasible and gives good yield [2]. Analytical grade reagents are used without further purification. 10 mmol of sodium Molybdate dihydrate ($Na_2MoO_4.2H_2O$) is dissolved in 43 ml of de-ionized water. 2ml of HCl is to be added drop-wise to thisand is continuously stirred using a magnetic stirrer. The solution thus prepared is poured into a stainless- steel autoclave with teflon lining (100ml) capacity, sealed, kept at 120 °C for 18 hours and cooled to room temperature. The resulting precipitate is washed with deionized water and ethanol many times and dried in vacuum at 60 °C for 24 hours.

2.2 Chemical method

250 ml of a 0.1 M ammonium molybdate tetrahydrate ($(NH_4)_6Mo_7O_{24}.4H_2O$), solution is mixed with 50 ml of ethylene glycol (99%). This solution is heated for 40 min at 120 °C, which produces a precipitate along with a dark blue solution. This mixture is cooled and

centrifuged to separate the precipitate. The precipitate is washed with deionized water, placed in the oven at 80 °C for a day. White, molybdenum trioxide powder is obtained which is sintered at a temperature of 700 °C in air [3]. The crystal structures of Molybdenum trioxide (MoO_3) along with unit cell, diffraction planes and Raman spectrum of are represented in **Fig.1-3**.

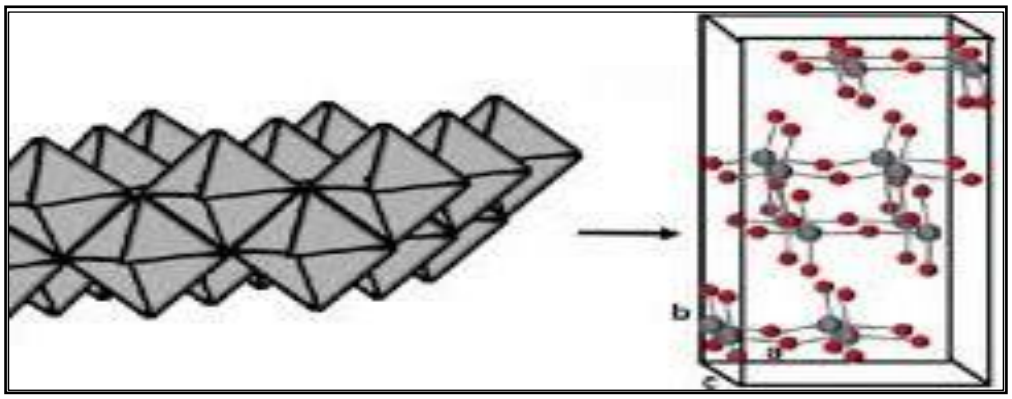

Fig.1 Crystal structure of ortho Molybdenum trioxide

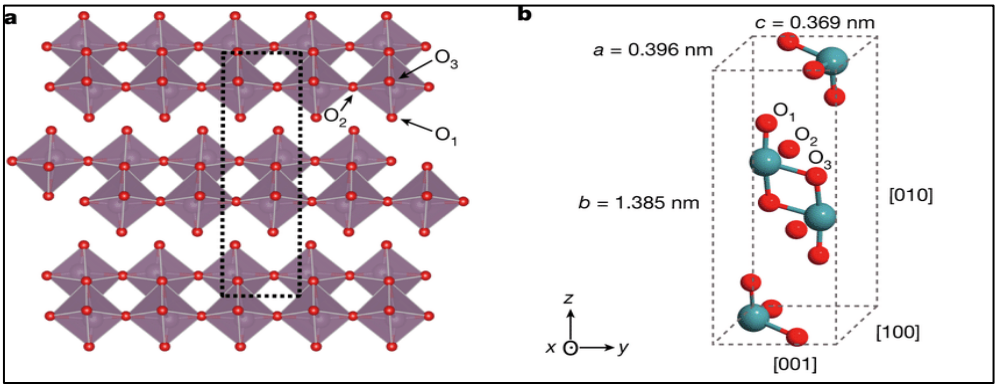

Fig. 2 (a) orthorhombic molybdenum trioxide structure (b) unit cell with lattice parameters

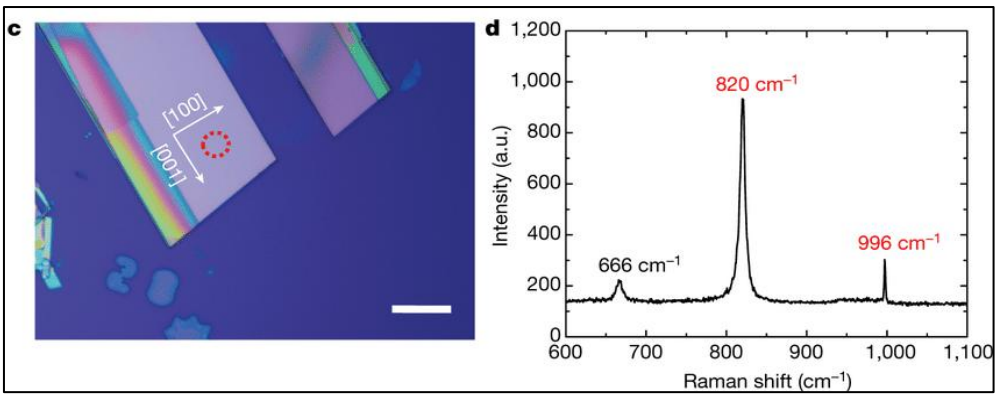

Fig. 3 (a) diffraction planes (b) Raman spectrum of ortho Molybdenum trioxide

3. Characterization

SEM, EDS and XRD are done for the prepared samples and the results are analysed [13]. XRD characterization was done with Rigaku Powder Xray diffractometer. In the same way SEM and EDS were done with APREO 2S from Thermo Fisher Scientific. The SEM and EDS image of MoO_3 by hydrothermal and chemical method are represented in **Fig. 4** and **5**. The percentage of elements in EDS spectrum of MoO_3 by hydrothermal and chemical method were tabulated in **Table 1** and **2**. The crystal grain sizes in MoO_3 by hydrothermal and chemical method were tabulated in **Table 3** and **4**.

3.1 Sample 1: SEM and EDS Molybdenum trioxide Prepared by hydrothermal method

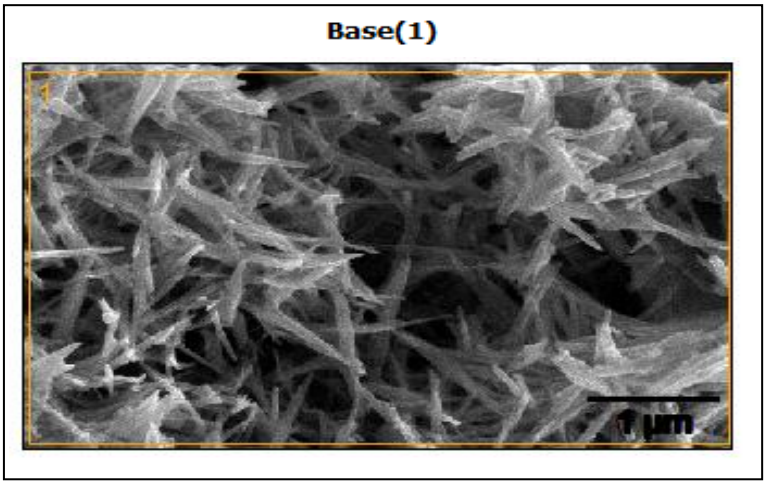

Fig. 4a SEM image of MoO_3

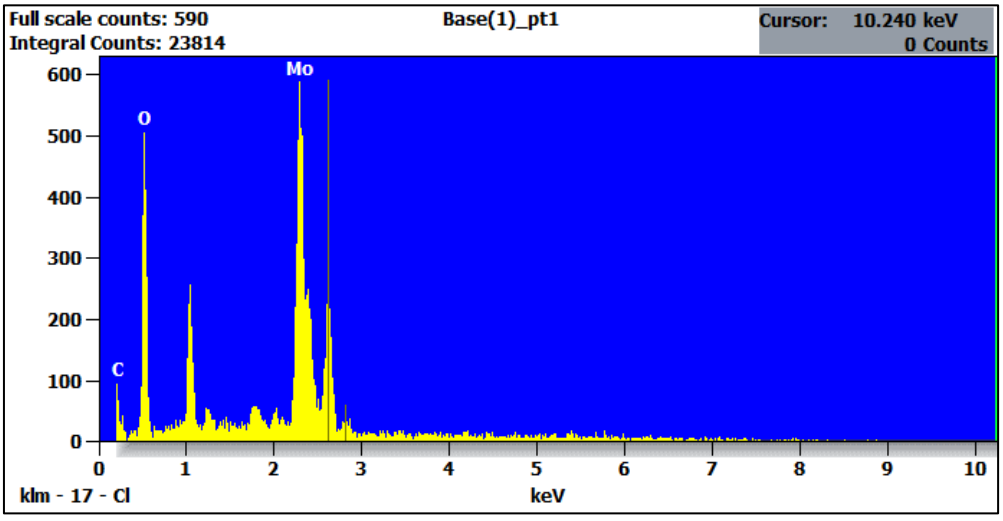

Fig. 4b EDS image of MoO_3

Table 1: Percentage of elements in EDS spectrum of MoO_3 by hydrothermal method

	C	***O***	***Mo***
Base(2)_pt1	981	21640	56852

Weight %

	C	***O***	***Mo***
Base(2)_pt1	0.87	37.32	61.81

Atom %

	C	***O***	***Mo***
Base(2)_pt1	2.37	76.50	21.13

Atom % Error (+/- 1 Sigma)

	C	***O***	***Mo***
Base(2)_pt1	±0.13	±0.62	±0.19

3.2 Sample 2: SEM and EDS Molybdenum trioxide Prepared by Chemical method

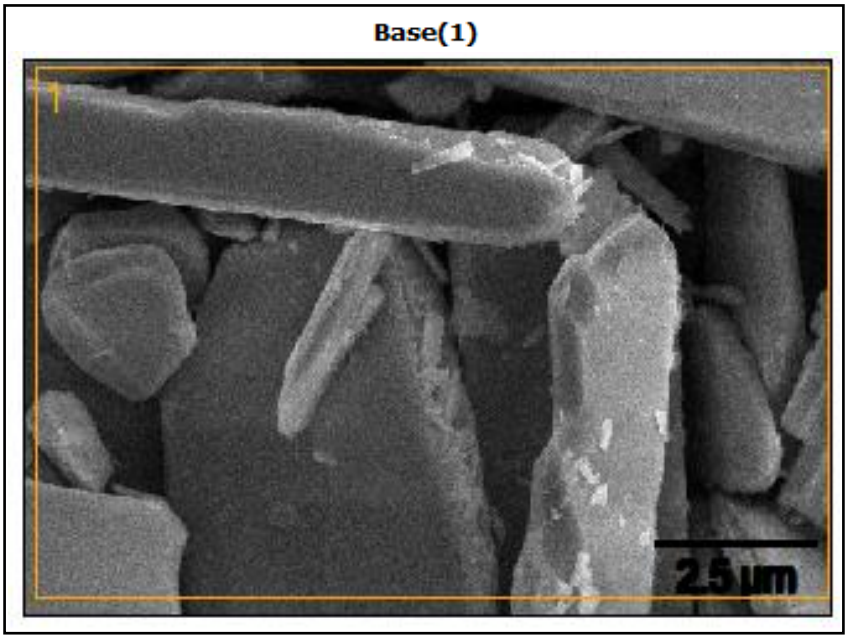

Fig. 5a SEM image of MoO_3

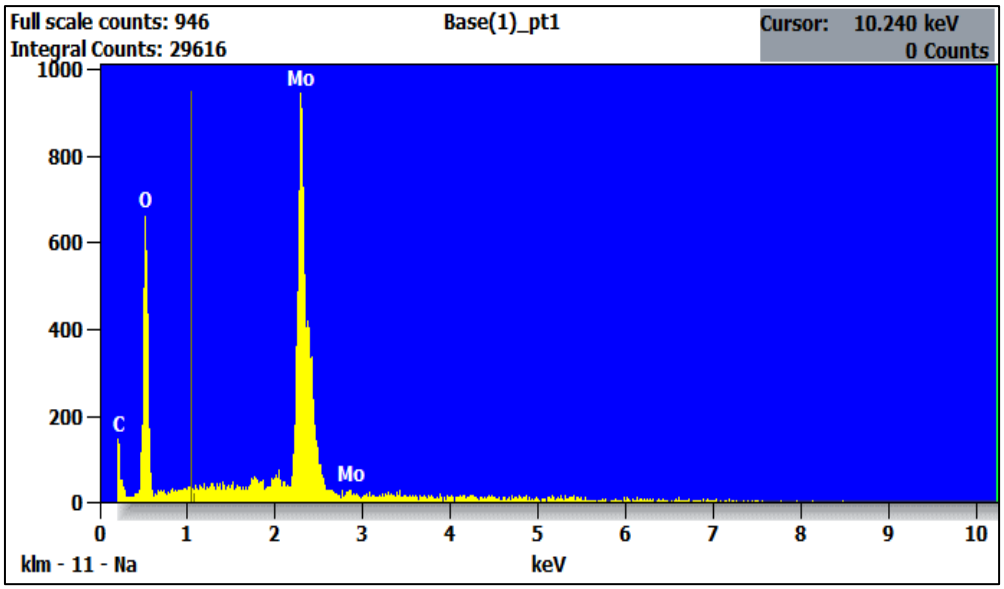

Fig. 5b EDS image MoO_3

Table 2: Percentage of elements in EDS spectrum of MoO_3 by chemical method

	C	***O***	***Mo***
Base(1)_pt1	341	3651	13634

Weight %

	C	***O***	***Mo***
Base(1)_pt1	1.43	30.93	67.63

Atom %

	C	***O***	***Mo***
Base(1)_pt1	4.33	70.11	25.56

Atom % Error (+/- 1 Sigma)

	C	***O***	***Mo***
Base(1)_pt1	±0.28	±1.19	±0.43

3.3 XRD of MoO_3 – hydrothermal method and chemical method

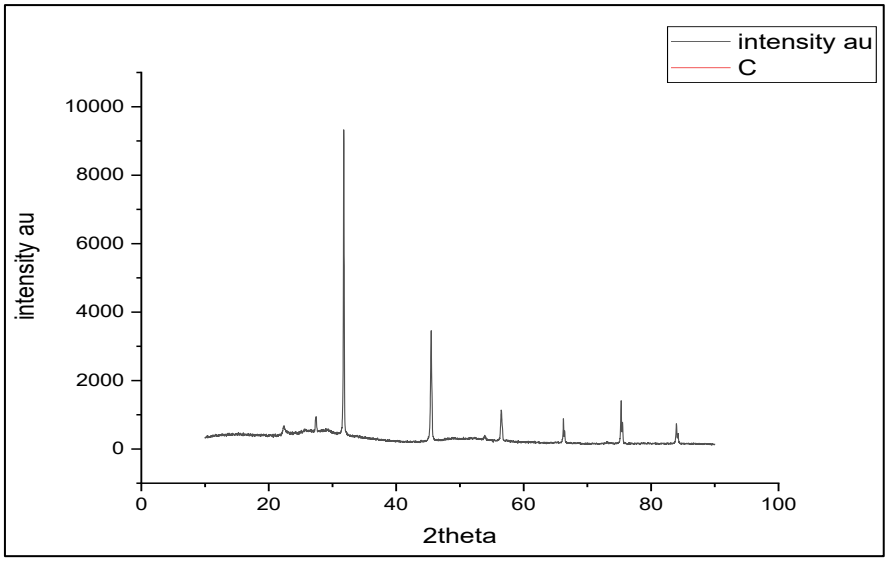

Fig. 6a XRD of MoO_3 by hydrothermal method

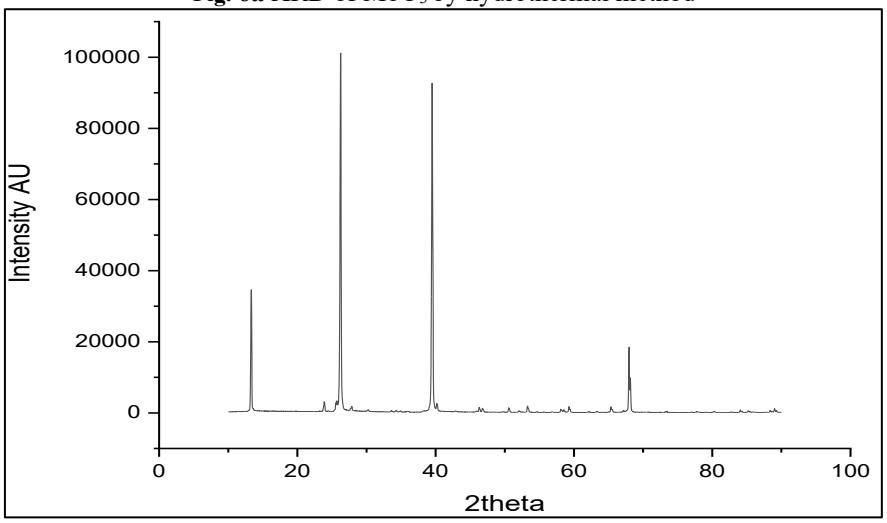

Fig. 6b XRD of MoO_3 by chemical method

Table 3: Crystal grain sizes in MoO_3 Hydrothermal method using Scherrer's formula

- $G=k\lambda/\beta\cos\theta$, where $k=0.89$ and $\lambda=1.54A^0$

2 θ	θ	Width	Grain size in nm
31.775	15.88	0.1611	50.69
45.488	22.74	0.2367	35.99

Table 4: Crystal grain sizes in MoO_3 chemical method using Scherrer's formula

- $G=k\lambda/\beta\cos\theta$, where $k=0.89$ and $\lambda=1.54A^0$

2 θ	θ	Width	Grain size in nm
13.40	6.70	0.1828	43.26
26.35	13.17	0.1826	43.86
39.50	19.75	0.1512	55.21
67.98	33.99	0.1205	78.60

4. Results and Discussions

The XRD pattern shows sharp peaks indicating that the nano materials obtained are crystalline. EDS pattern shows consistent weight percentage of elements. SEM pattern shows that the nano materials have the morphology of nano belts.The diffraction peaks demonstrated the crystal plane growth. Several characteristic diffraction peaks at 2θ corresponding to 020, 040, 060, 042, 081, 0100, planes. Accordingly alpha MoO_3 belts and nanobelts grew with a specific orientation. Crystal grain sizes were determined using Sherrer's formula, $G=k\lambda/\beta\cos\theta$. All the peaks correspond with MoO_3 JCPDS reference card 36 - 0609. Diffraction pattern shows high crystallinity, orthorhombic and pure phase of alpha MoO_3. The peaks obtained correspond to Mo- and O-atoms present in the nanobelts. The EDS spectra showed the existence of 'Mo' and 'O' with a molar ratio close to 1:3. The results confirmed and correlated with a stoichiometric mole ratio for the formation of MoO_3 with nanocrystalline structure.The Sample was composed of 'Mo' and 'O' only with not much traces of secondary impurities.

5. Hydrothermal and chemical methods: A comparative study

Hydrothermal method is feasible but difficult to handle with its autoclave. But its yield is good. Chemical method uses simple glass wares but the yield is less. Both hydrothermal and chemical method gives very sharp peaks for XRD. Crystal grain sizes are slightly larger for chemical method. The EDS spectrum is purer for chemical method. SEM represent morphology of nanobelts in both the methods. And the nanobelts obtained are highly crystalline.

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