

# PHOTOCHROMIC PROPERTY OF DMF INDUCED M<sub>0</sub>O<sub>3</sub>NANO POWDER USING SOLGEL METHOD

Saieswari G<sup>1</sup>, M.Naveen Rooba Doss<sup>2</sup>, R ilavarasi<sup>3</sup>

Nanotechnology Division, Department of Electronics and Communication Engineering,

PeriyarManiammai Institute of Science and Technology, Thanjavur, India.

## ABSTRACT

In the present research, the photochromic  $MoO_3$  powder has been synthesized via simple sol gel, using Dimethylformamide. The Dimethyl formamide-induced  $MoO_3$  powder is found to have enhanced photochromic properties. The influence of DMF induced  $MoO_3$  microstructure and photochromic properties was carried out by scanning electron microscopy(SEM) and particle size analyze were determined by DLS. The sample turned Blue color when exposed to UV visible light irradiation for 30 minutes. The colored films bleached to the colors initial after 8 h.

Key words: MoO3, DMF, Photochromism, Nanopowder, Irradiation

#### 1 Introduction

Photochromic material, which is change color upon light irradiation, refers to the persistent and reversible transformation between different excited states[1].photochromic material have been investigated into two groups: organic(e.g., azobenzene , spiropyran and diarylethene) and inorganics (e.g., transition metal oxide) and these photochromic material were used for various applications. Inorganic photochromic material exhibit good stability and cost effective while compare to organic material. Photochromic transition material include molybdenum oxide (MoO<sub>3</sub>), titanium dioxide (TiO2),tungsten oxide (WO<sub>3</sub>),Vanadium pentoxide (V<sub>2</sub>O<sub>5</sub>), niobium pentoxide (Nb<sub>2</sub>O<sub>5</sub>) and ZnO, Upon band gap irradiation electron hole pair can generate in transition metal oxide[2].

As wide band gap of n type semiconductor material MoO<sub>3</sub> are attracting much attention as both photochromic and electro chromic property due to high optical contrast and reversible color change[3,4]. The transition metal oxide MoO<sub>3</sub> is multifunctional interesting material in various field of application such as smart windows, optical switches, chemical sensor and storage media, optical fiber, fuel cells, display devices [5,4]. Basically MoO<sub>3</sub> has three crystallinepolymorphs: thermodynamically stable orthorhombic phase  $\alpha$ -MoO<sub>3</sub> has orthorhombic crystal structure, meta stable  $\beta$  –MoO<sub>3</sub> has cubic rhenium trioxide (ReO<sub>3</sub>) and metastable high pressure phase hexagonal h- MoO<sub>3</sub>[7-9].



MoO<sub>3</sub> have been synthesized by various chemical method[10], such as chemical vapor deposition[11], sol gel [12],HCL chemical precipitation[13], thermal evaporation[14], electrodeposited[15], hydrothermal[16], electrochemical processing[17]. Among this sol gelmethod is the most promising solution based chemical method. It is used to synthesis all kind of nanostructured material and advantage of solgel method is easy to control over the crystal structure (MoO<sub>3</sub>, MoO<sub>3</sub>,h- MoO<sub>3</sub>),the morphology and size by different reaction parameter reaction time, reactant solvent medium, reactant source, reaction temperature, additives and also lowcost, flexibility and simplicity[18,19].

 $MoO_3$  crystal powder exhibits various morphologies such as nanotubes,hollow  $MoO_3$ nanospheres, nanorods, Mesostructuredtoroids, MoO3 fiber and nanobelts[20]. In the present research, the photochromic  $MoO_3$  powder has been synthesized via a simple sol gel method, using Dimethylformamide as the capping agent. The Dimethyl formamide-induced  $MoO_3$  powder is found to have enhanced photochromic properties.

However the drawback of  $MoO_3$  used in photochromic material consequently poor reversibility of coloration and structural instability[21]. While using DMF organic inducer the photochromic properties of  $MoO_3$  have been increased and it is highly bio compatible too. The chromogenic properties of  $MoO_3$  that is the ability to change the optical density. It is due to the formation of color center changes when exposed to external agents. The external agent is light, then the material named photochromic and color changes due to external agent heat named thermochromic[22].

DMF is a clear liquid widely used in industries as a solvent reagent and catalyst, additive or an intermediate. As shown in et al[17] formaldehyde induced  $MoO_3$  exhibit hierarchical mesoporous as well as photochromic property. Thus ,in this work motivated to investigate the photochromic properties of DMF induced  $MoO_3$ sol gel method and related mechanism of photocoloration.

#### 2. Experimental

Molybdenum oxide nanoparticle were prepared using solgel method. Certain amount of sodium molybdate  $(Na_2MoO_4 \cdot 2H_2O)$  was dissolved in 25 ml deionized water to get the transparent solution of 1.0Msolution. 10 ml of Dimethyl formamide (DMF) solvent reagentwas added drop wise into the solution under constant stirring at room temperature. Then the pH of above mentioned solution was adjusted to be 1.0 using concentrated hydrochloric acid (HCL) solution by simultaneous vigorous agitation for 4h in succession. The precipitates were filtered and washed several times in deionized water and ethanol to avoid impurities of the end product. After that the white precipitates was transferred into petri dish to dry the sample in a hot air oven at 90°C for 1 hr. Initially xerogel and final white powder was obtained. The powder was then cured under UV lamp for photochromic property.





Scheme 1 :Schematic representation of step wise synthesis process of MoO<sub>3</sub>nano powder and photochromic property



## 3. Test ForPhotochromism

Scheme 2: Experimental setup of photochromic property of powder sample



UV colored MoO3 powder are shown in figure 1 the virgin MoO<sub>3</sub> powder are white transparent in the visible region and it turns to blue under UV light irradiation. The mechanism of coloration in MoO3 under the irradiation of UV light with photon energies greater than MoO<sub>3</sub>band gap, electron can excited to the conduction band, leaving a hole in the valence band. The photochromic property of MoO<sub>3</sub>Nano powder sample was tested by mercury halogen lamp. The prepared sample was irradiated under UV lamp (10cm away from the sample,360nm,3W) for 30min. Initially the color of sample was white transparent and the transparency state was changed to colored state under irradiation of UV light for 1 hour, and after the sample was kept in a dark place the colored state partially bleached back to the initial state.





## 4.Characterization

The as prepared MoO<sub>3</sub>nano powder morphology was determined by scanning electron microscope and the morphology of final product is considered to play a vital role in the photochromic properties of MoO<sub>3</sub>nano powder and the presence photochromic properties were tested by using UV light radiation.

Figure 2 shows the SEM image of the synthesized DMF induced MoO3 powder. MoO3 powder with DMF exhibit nearly like hierarchical mesoporous spherical morphology .The inducer does not have great effect on morphology and particle size distribution was non uniform and large of aggregation formed.MoO<sub>3</sub> nucleation and subsequent growth does not occur due to loss of temperature.



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Figure 2.SEM image of DMF induced MoO3 powder





Dynamic light scattering (DLS) which is also known as quasi-elastic light scattering or photon correlation spectroscopy , is a technic used for size distribution and particle size studies. Commonly the dispersion and stability is an essential factor in view of preparing nanoparticle. The DLS measurement taken from the DMF induced MoO<sub>3</sub>nanopowder was present in figure 2. The presence of MoO<sub>3</sub>nano powder disperses uniformly and narrows size distribution. Furthermore the diameter of the particle is 149.3nm and the polydispersity index value is 0.252 respectively, So it indicates the narrow distribution of particle size.



# 5.Conclusion

Molybdenum trioxideNanopowder synthesized using a simple sol gel method and the presence of reversible photochromicproperties of the powder were observed under UV irradiation. The DLS showed the uniformly narrow size distribution of MoO<sub>3</sub>Nano powder sample and the result of SEM the morphology of MoO<sub>3</sub> Powder nearly exhibit hierarchical mesoporous spherical. The powder showed a considerable fast response to coloring 30 min. The bleaching process takes a longer time to return to their initial state, approximately 8 hours.

### Reference :

[1] Miyazaki, Hidetoshi, Takahiro Matsuura, and Toshitaka Ota, TiO2 Nano-Particles Based Photochromic Composite Films, *Composites Communications* 10 (2018):136–39.

[2]<u>Shufen Wang, WeirenFan,Zichuan Liu, Aibing Yu</u> and <u>XuchuanJiang</u>,Advances on Tungsten Oxide Based Photochromic Materials: Strategies to Improve Their Photochromic Properties, *Journal of Materials Chemistry C* 6(2) (2018) :191–212.

- [3]Shen, Yi, Rong Huang, Yuanyuan Cao, and Pingping Wang. 2010. "Synthesis and Photochromic Properties of Formaldehyde-Induced MoO 3 Powder." *Materials Science and Engineering B: Solid-State Materials for Advanced Technology* 172(3): 237–41. http://dx.doi.org/10.1016/j.mseb.2010.05.023.
- [4] Taurino, Antonella M. et al. 2006. "Synthesis, Electrical Characterization, and Gas Sensing Properties of Molybdenum Oxide Nanorods." *Applied Physics Letters* 88(15): 1–4.
- [5]Pereira, L. G. et al. 2008. "Influence of Doping on the Preferential Growth of α-MoO3." *Journal of Alloys and Compounds* 459(1–2): 377–85.
- [6]Sun, Mu et al, "Nanocrystalline Tungsten Oxide Thin Film: Preparation, Microstructure, and Photochromic Behavior." *Journal of Materials Research* 15(4) (2000): 927–33.
- [7]Krishnan, Chirakkal V. et al. 2009. "Formation of Molybdenum Oxide Nanostructures Controlley by Poly(Ethylene Oxide)." *Chinese Journal of Polymer Science (English Edition)* 27(1): 11–22
- [8]Chithambararaj, A., N. Rajeswari Yogamalar, and A. Chandra Bose. 2016. "Hydrothermally Synthesized H-MoO3 and α-MoO3 Nanocrystals: New Findings on Crystal-Structure-Dependent Charge Transport." *Crystal Growth and Design* 16(4): 1984–95.
- [9]Navas, I. et al. 2009. "Effect of Zinc Oxide Doping on the Structural and Optical Characterization of Nanostructured Molybdenum Oxide Films." *Journal of Nanoscience and Nanotechnology* 9(9): 5254–61.



- [10]Chiang, Tzu Hsuan, and Hung Che Yeh. 2013. "The Synthesis of α-MoO3 by Ethylene Glycol." Materials 6(10): 4609–25.
- [11]Ivanova, T., M. Surtchev, and K. Gesheva. 2002. "Investigation of CVD Molybdenum Oxide Films." *Materials Letters* 53(4–5): 250–57.
- [12]Dong, Winny, and Bruce Dunn. 1998. "Sol-Gel Synthesis of Monolithic Molybdenum Oxide Aerogels and Xerogels." *Journal of Materials Chemistry* 8(3): 665–70.
- [13]Mizushima, Takanori, Kazuya Fukushima, Hironobu Ohkita, and Noriyoshi Kakuta. 2007. "Synthesis of β-MoO3 through Evaporation of HNO3-Added Molybdic Acid Solution and Its Catalytic Performance in Partial Oxidation of Methanol." *Applied Catalysis A: General* 326(1): 106–12.
- [14]Kalantar-Zadeh, Kourosh et al. 2010. "Synthesis of Nanometre-Thick MoO3 Sheets." *Nanoscale* 2(3): 429–33.
- [15]Yao, David Di et al. 2012. "Electrodeposited α- And β-Phase MoO 3 Films and Investigation of Their Gasochromic Properties." Crystal Growth and Design 12(4): 1865–70.
- [16]Yang, Xiaofei et al. 2011. "NaCl-Assisted Hydrothermal Synthesis of High-Quality Crystalline α-MoO3 Nanobelts." Crystal Research and Technology 46(4): 409–12.
- [17]McEvoy, Todd M., Keith J. Stevenson, Joseph T. Hupp, and Xiaojun Dang. 2003. "Electrochemical Preparation of Molybdenum Trioxide Thin Films: Effect of Sintering on Electrochromic and Electroinsertion Properties." *Langmuir* 19(10): 4316–26.
- [18]Shen, Yi et al. 2014. "Effect of Formaldehyde on the Photochromic Properties of Ordered Molybdenum Oxide Thin Films Produced by Hydrothermal Process." Surface and Coatings Technology 240: 393–98. <u>http://dx.doi.org/10.1016/j.surfcoat.2013.12.062</u>
- [19]Jittiarporn, Phuriwat et al. 2016. "Synthesis of H-MoO<sub>3</sub> and (NH<sub>4</sub>)<sub>2</sub>Mo<sub>4</sub>O<sub>13</sub> Using Precipitation Method at Various PH Values and Their Photochromic Properties." *Applied Mechanics and Materials* 835: 34–41.
- [20]Dai, Z. R., J. L. Gole, J. D. Stout, and Zhong L. Wang. 2002. "Tin Oxide Nanowires, Nanoribbons, and Nanotubes." *Journal of Physical Chemistry B* 106(6): 1274–79.
- [21]Zhang, Yuzhi et al. 2008. "Photochromic Behavior of Li-Stabilized MoO3 Sol-Gels." *Journal of Non-Crystalline Solids* 354(12–13): 1276–80.
- [22]Arvizu, M. A. et al. 2012. "Photochromism and Thermochromism of MoO 3 Thin Films Doped with ZnSe." *AIP Conference Proceedings* 1420(1): 151–56.