

# Preparation and Characterization of Nanohybrid of MoO<sub>3</sub> through Different Methods

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## Abstract

The synthesis and characterization of an organic-inorganic hybrid compound (MoO<sub>3</sub>.PABA) is presented. This nanostructured hybrid characterized by elemental analysis, infrared spectroscopy, thermogravimetric study, X-ray powder diffraction analysis and scanning electron microscopy. It verified that this compound consists of two-dimensional chains of molybdenum oxides in which organic ligands directly coordinated to the oxide layers. This nanohybrid is a new form of molybdenum oxide hybrid with a good potentials of being employed as an efficient catalyst in oxygen atom transfer reactions.

**Keywords:** Molybdenum Oxide; Hybrids; Hydrothermal; X-ray Diffraction; Nanocatalysts

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## 1. Introduction

Inorganic-organic hybrid materials, especially metal oxides are the main concern of many current research projects, owing to their potential applications in catalysis, sorption, photochemistry and electronics [1-3]. The synthesis of inorganic-organic hybrids by intercalation of organic ligands into layered molybdenum oxide have been extensively explored [4-16]. These hybrids could exhibit the properties of both, the inorganic and organic phases. Organic components act as a ligand incorporated into the metal oxide backbone. It is recognized that organic component can influence the structure of the inorganic oxides in the resultant composites. Furthermore, such organic-inorganic hybrid materials have a new properties arising from the interplay of two components.

In this work we describe our attempts to synthesize new molybdenum oxide nanohybrid by intercalation of 4-aminobenzoic acid (PABA) into molybdenum oxide layers.

This nanostructured hybrid is well characterized by elemental analysis, infrared spectroscopy, thermogravimetric study, SEM and X-ray powder diffraction analysis. This nanohybrid is a new form of molybdenum oxide hybrid with a good potential of being employed as an efficient catalyst in oxygen atom transfer reactions.

## 2. Experimental

### 2.1 Materials and Methods

All chemicals were used as received without further purification. Heroeus CHNO-Rapid analyzer is used for elemental analyses. Infrared spectra are recorded on a Bruker Equinox55 spectrometer. Thermogravimetric analysis are carried out using PL STA 1500 system under air atmosphere with a heating rate of 10°C min<sup>-1</sup>. The X-ray diffraction analysis are obtained in Philips XRD equipment using Cu K $\alpha$  radiation ( $\lambda = 1.54060 \text{ \AA}$ ). SEM images are carried out using Philips XL300 instrument.

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## 2.2 Synthesis of (MoO<sub>3</sub>.PABA)

A mixture of 1mmol of yellow molybdic acid and 1mmol of 4-aminobenzoic acid (PABA) along with 20 ml of solution containing ethanol and water in a 1:3 ratio was stirred for approximately 48h, until a gray solid powder was obtained. The product was collected by filtration and dried at room temperature (yield ca. 78% based on molybdenum).

## 2.3 Catalytic tests

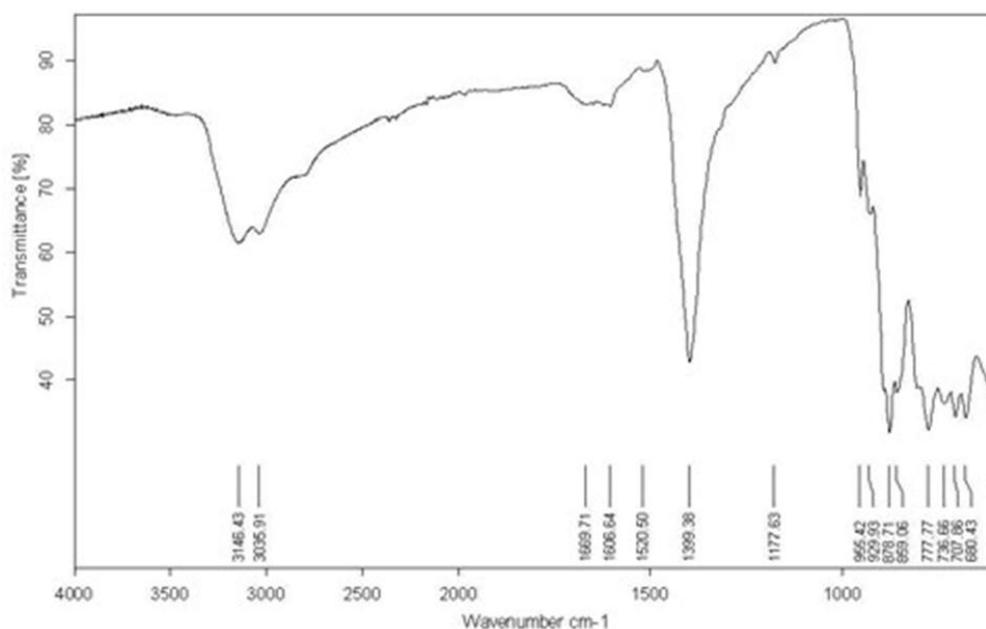
The catalytic activity was examined by suspending 1.5 % molar ratio (cat / substrate) (0.11 mmol) catalyst in 1.5 ml CH<sub>2</sub>Cl<sub>2</sub> and 3.65 mmol of pre-selected alkenes. Hydrogen peroxide 30 % (1 ml) was introduced to the reaction mixture. The two phase reaction system was stirred in room temperature. The substrate and reaction products

are dissolved in organic phase and catalyst is presented in water phase. The extent of alkenes conversion was monitored by sampling aliquots of organic phase of reaction mixture every half an hours and analyzed by gas chromatograph. After the reaction completed, the water phase easily filtered to remove any suspended materials and can be repeatedly used as a recyclable catalyst.

## 3. Results and Discussion

### 3.1 characterization of nanohybrid

Figure 1. shows the FT-IR spectrum of (MoO<sub>3</sub>.PABA) in the range 400-4000 cm<sup>-1</sup> at room temperature. The strong band at 955cm<sup>-1</sup> assigned to  $\nu(\text{Mo}=\text{O})$ , the strong band at 878 is attributed to  $\nu(\text{Mo}-\text{O}-\text{Mo})$ , bands at 777 and 589 cm<sup>-1</sup> are assigned to  $\nu(\text{O}-\text{Mo}-\text{O})$ , and the other bands are due to vibration modes of organic ligands



**Figure 1.** IR spectra of (MoO<sub>3</sub>.PABA) compound

Molybdenum oxide nanostructures with various morphologies were obtained using a novel route at ambient conditions. Yellow molybdic acid has a monoclinic crystal structure with cell dimensions of  $a=10.476 \text{ \AA}$ ,  $b=13.833 \text{ \AA}$ ,  $c=10.606 \text{ \AA}$  and  $\beta=91.63^\circ$ . It adopts a layered structure where the distorted layers are constructed by corner sharing MoO<sub>5</sub> octahedra. The layers are stacked along the b axis and interconnected by interlayer water molecules.

By replacing the interlayer water molecules under mild condition with organic ligands a control over the morphology can be gained.

The X-ray diffraction patterns of the synthesized hybrid is shown in figure 2. As can be observed, the XRD pattern exhibit sharp intense reflection and a gradual increase in the  $d$  spacing of the (001) reflection is observed depending on the organic ligands.

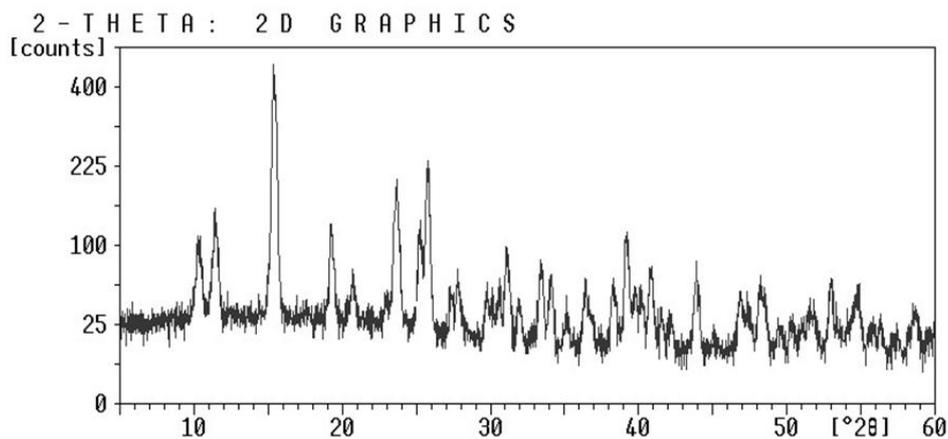


Figure 2. X-ray diffraction pattern of (MoO<sub>3</sub>.PABA)

The TGA curve of the hybrid material shows a weight loss occurred at about 460°C, which is due to the dissociation of organic ligands which is agreed with the calculated value.

Figure 3 shows a SEM image of hybrid material (MoO<sub>3</sub>.PABA). The SEM image shows particles varying in size from approximately 60-200 nm. Each particle is composed of multiple lamellar sheets.

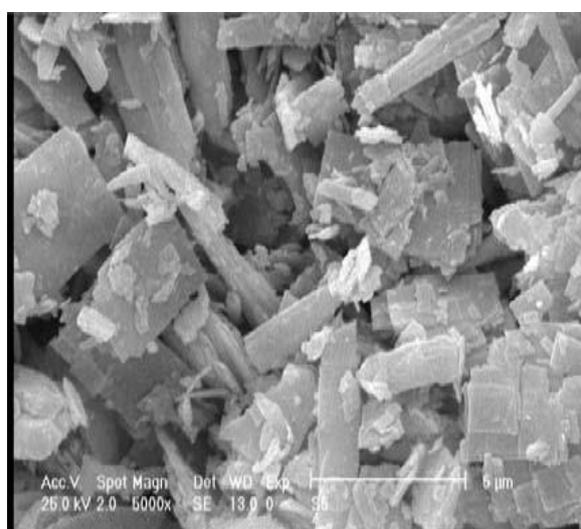


Figure 3. SEM image of (MoO<sub>3</sub>.PABA)

### 3.2 Catalytic performance

Figure 4 shows conversion in epoxidation of cyclohexene and cyclooctene at room temperature as function of time with selectivity above 99%. As can be seen conversion is quite low in beginning of the reaction and reaches to a plateau after about 6 hours. It shows that our nanocatalyst could successfully catalyzed the epoxidation of the selected olefins with high reactivity and selectivity. Therefore, it is recommended as a suitable catalyst for epoxidation of olefins.

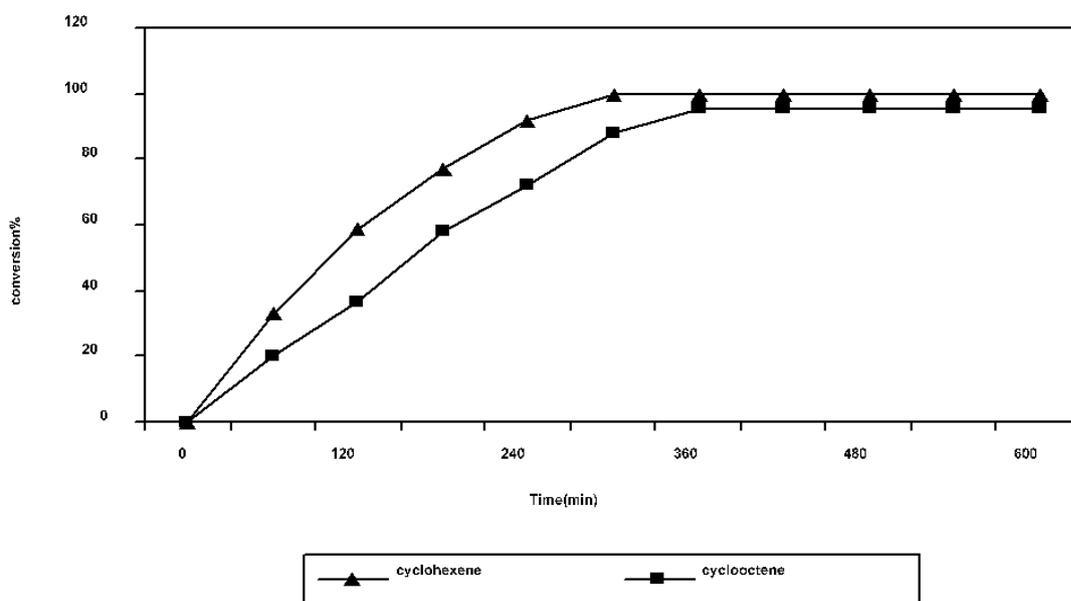


Figure 4. Epoxidation of cyclooctene and cyclohexene at room temperature

#### 4. Conclusion

The intercalation of organic ligands into oxide layers in mild condition is led to the successful syntheses of many molybdenum oxide hybrids. Therefore, our aim was to synthesize two dimensional molybdenum oxide hybrid based on MoO<sub>3</sub> layers with 4aminobenzoic acid directly coordinated to the oxide layers.

Results show that this nanohybrid material has a good potential of being employed as an efficient catalyst in epoxidation of alkenes.

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