**Supporting Information**

# Green synthesis and characterization of anisotropic uniform single-crystal α-$MoO_3$ nanostructures

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**The determination of the nominal formula of the sample obtained at 81.5 °C**

Under vacuum conditions, molybdenum peroxo complex is decomposed into molybdenum trioxide, water, and oxygen from about 147 °C to around 257 °C [1].

The decomposition of the sample obtained at 81.5 °C could be shown in Equation (1):

$$MoO_{3-x}(O_2)_x \cdot yH_2O \xrightarrow{\Delta} MoO_3 + \frac{x}{2}O_2 + yH_2O \quad (1)$$

The weight loss of $O_2$ and the total weight loss could be shown in Equation (2) and (3) respectively:

$$\text{Weight loss } (O_2)\,\% = \frac{\frac{x}{2}M_{O_2}}{M_{MoO_{3-x}(O_2)_x \cdot yH_2O}} \times 100\% \quad (2)$$

$$\text{Total weight loss } (O_2 + H_2O)\,\% = \frac{\frac{x}{2}M_{O_2} + yM_{H_2O}}{M_{MoO_{3-x}(O_2)_x \cdot yH_2O}} \times 100\% \quad (3)$$

where $M$ is the molecular weight. From the weight loss of $O_2$ and total weight loss, the value x and y in the nominal formula could be calculated.

Temperature programmed decomposition-mass spectrometry (TPDE-MS) and the external standard method were carried out in a quartz microreactor coupled to an Omnistar™ GSD 301 O3 mass spectrograph. The carrier gas used was $N_2$ at a flow rate of 25 mL·min$^{-1}$.
Before the TPDE-MS investigation, about 0.2000g sample obtained at 81.5 °C was heated at 70 °C until the $O_2$ signal had been calm and been held under $10^{-13}$. When TPDE was carried out with a 15 °C·min$^{-1}$ heating ramp, the $O_2$ (m/e=32) profile was recorded.
In the external standard method, the injection was performed before the blank quartz reactor which was kept at 200 °C.

All the calculations concerning the quantitative analysis were performed with the external standard by measurement of the peak areas. The peak area of each dose of $O_2$ was plotted against the volume to obtain the calibration graph. The 6 volumes were subjected to regression analysis to calculate the calibration equation and correlation coefficient. Linearity was obtained between the peak area and the volume of 0.5-5.0 mL with $R^2$=0.99962 for $O_2$.

Equation: $y = 2.56757\times10^{-9} \cdot x - 1.694\times10^{-10}$

where y means the peak area, and x means the volume (mL).

The volume of $O_2$ was about 4.6 mL (standard temperature and pressure), equal to about 3.3% of weight loss.

From the weight loss of $O_2$ (3.3%) and total weight loss (11.6%), the value x (0.33) and y (0.75) in the nominal formula could be calculated.

**Reference:**

[1] Segawa, K.; Ooga, K.; Kurusu, Y. *Bull. Chem. Soc. Jpn.* **1984**, *57*, 2721

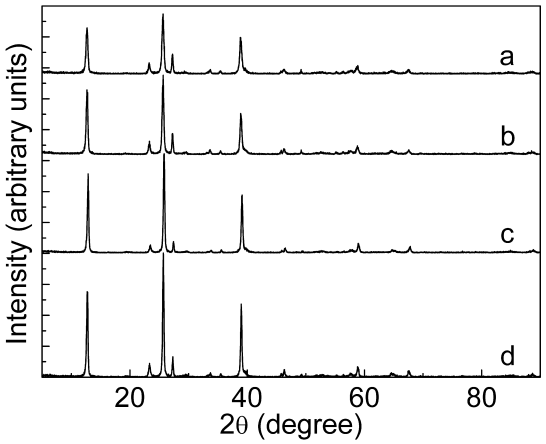


**Figure S1.** XRD patterns of solid products prepared from the 0.9 mol/L molybdenum solution at 170 °C for different time: (a) 1 h, (b) 5h, (c) 20h, and (d) 45h (Figure S1d is identical with Figure 2e).