

Supporting Information

Preparation of MoO₃ QDs through combining intercalation and thermal exfoliation

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1. Characterization of MoO₃ commercial powder and MoO₃-amine composites

The successful intercalation of MoO₃ by n-butylamine to prepare MoO₃-amine composites was confirmed by SEM, XRD and IR spectra.

Before intercalation, commercial MoO₃ powder possessed layered structure (Fig. S1a and S1b). Its XRD spectrum (Fig. S2a) matched well with the orthorhombic MoO₃ (JCPDS card No. 05-0508) and showed the (020) peak at 12.8° (the corresponding space between adjacent layers was 6.9 Å). After intercalation, the formed MoO₃-amine composites still showed layered structure, but were much ambiguous (Fig. S1c and S1d), indicating the composites have low crystallinity. This was verified by its corresponding XRD spectrum, since there was only one broad peak located at 6.3°, corresponding to the interlayer space of 13.9 Å which was a little smaller than the reported value.

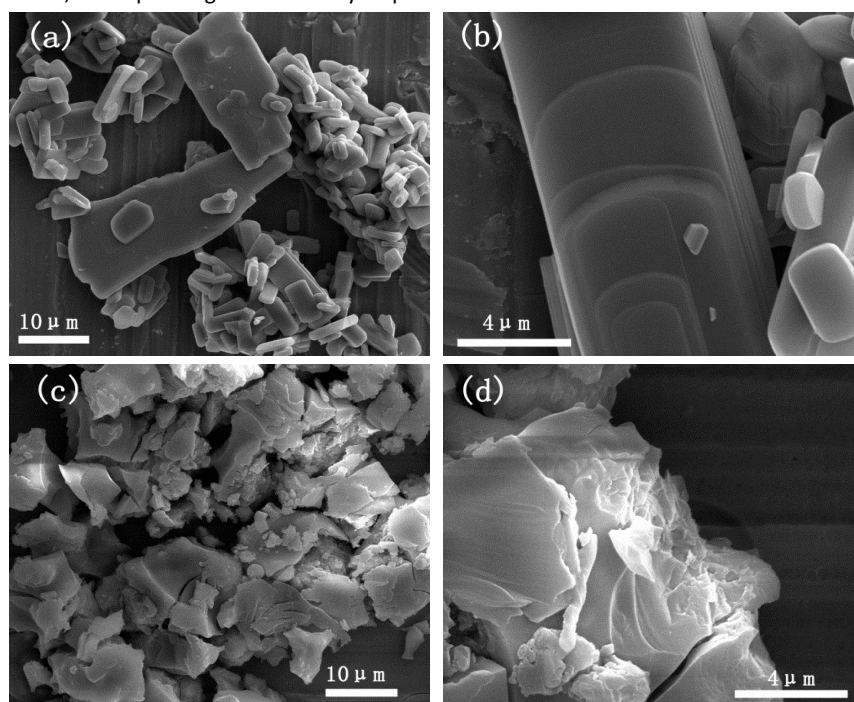


Fig. S1 The SEM images of MoO₃ commercial powder (a) (b) and the MoO₃-amine composites obtained after intercalation (c) (d).

For the IR spectrum of commercial MoO₃, the molybdenum oxide lattice vibrations appeared at 594, 868 and 995 cm⁻¹, respectively (Fig. S2). After intercalation, because of the presence of alkylamines, the corresponding alkylamines bands arose. Two bands appeared at 2870 and 2960 cm⁻¹ could be attributed to the antisymmetric and symmetric -CH₂ stretching vibrations, respectively. Bands corresponding to -NH bending vibration and stretching vibration were observed at 1610 and 3440 cm⁻¹. And at the same time, because of the interaction between the N-H from the amine and the Mo-O from the lattice, by means of a hydrogen bond, the vibration bands of MoO₃ all shifted to lower wave numbers as compared with the bands of commercial MoO₃.

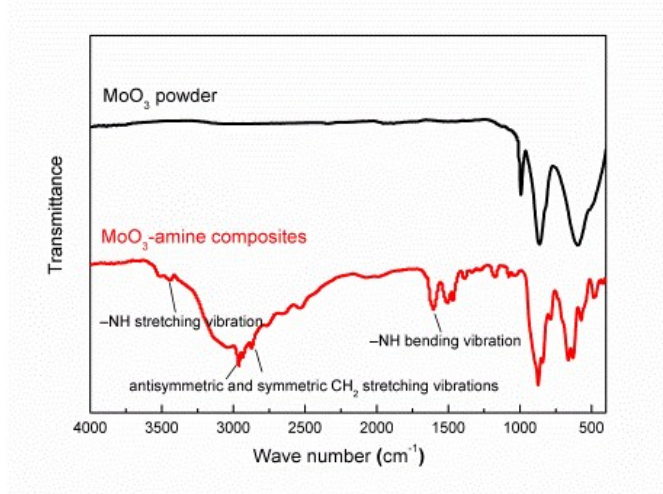


Fig. S2 The IR spectra of MoO₃ commercial powder and the MoO₃-amine composites.

2. Characterization of the products left after thermal exfoliation under 460 °C for different time

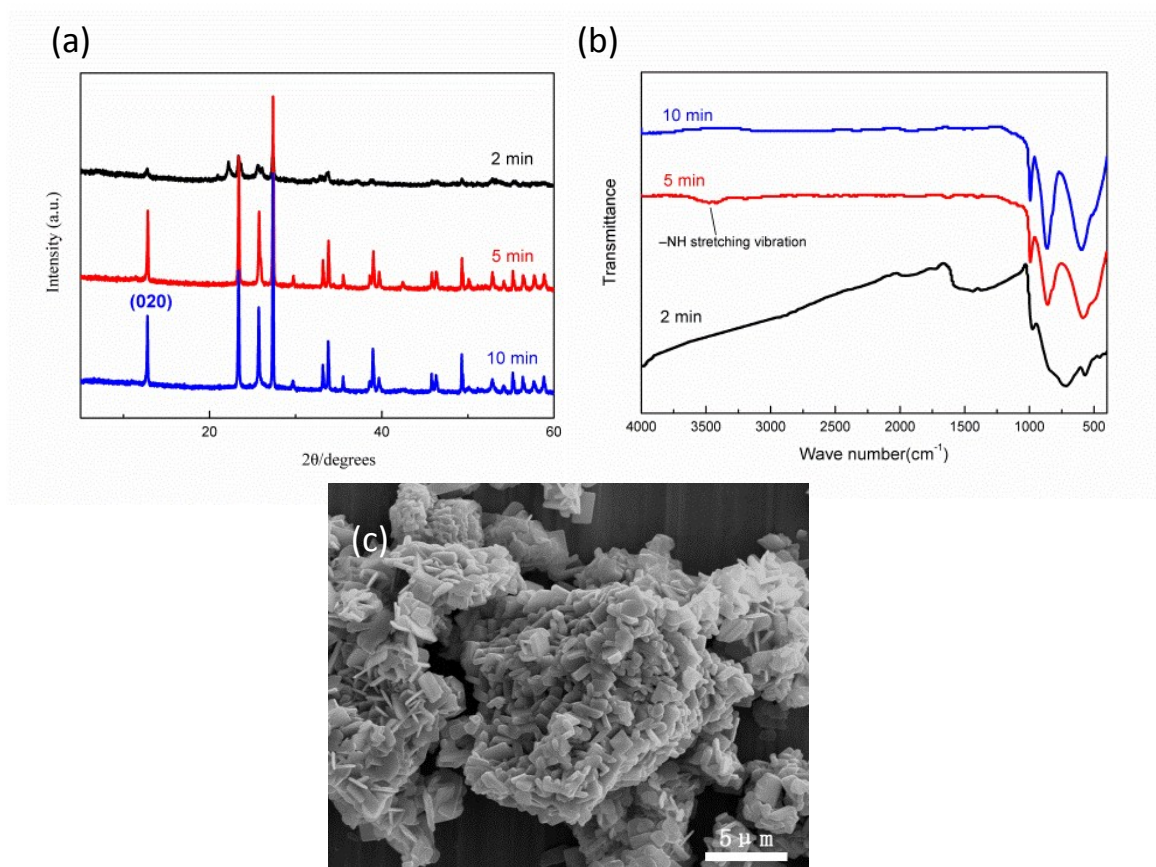


Fig. S3 The XRD patterns (a) and their corresponding IR spectra (b) of the products left after thermal exfoliation

under 460 °C for different time. The SEM image (c) of the products left after thermally treating under 460 °C for 10 min showed remarkable size decreasing compared to commercial MoO₃ powder as the outcome of the fragment of MoO₃ layers under the drastic increment of pressure.

3. QDs detached from rough MoO₃ particles after sonication

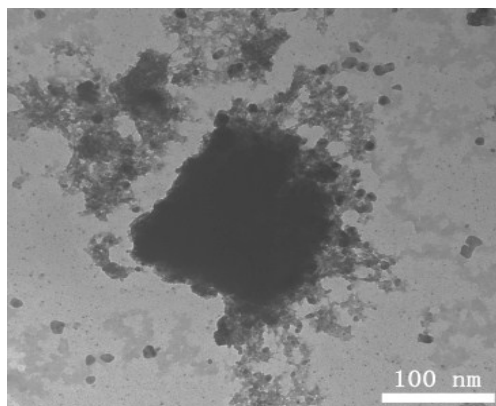


Fig. S4 The TEM image of particles obtained after sonication (thermal exfoliation under 460 °C for 10 min) showed that small particles including QDs were detached from rough MoO₃ particles.

4. Direct sonication of ground MoO₃ powder

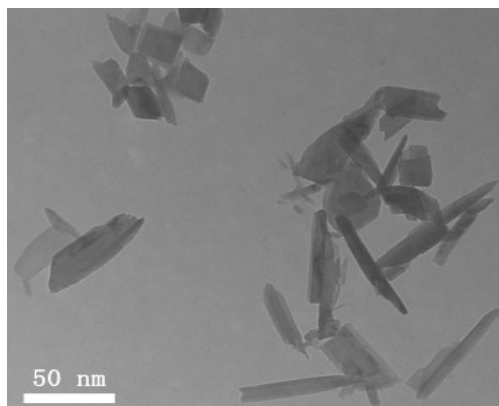


Fig. S5 The TEM image of MoO₃ nanosheets obtained after direct sonication of ground MoO₃ powder. No QDs were found after direct sonication.

5. Contrast experiments conducted under 530°C and 600°C

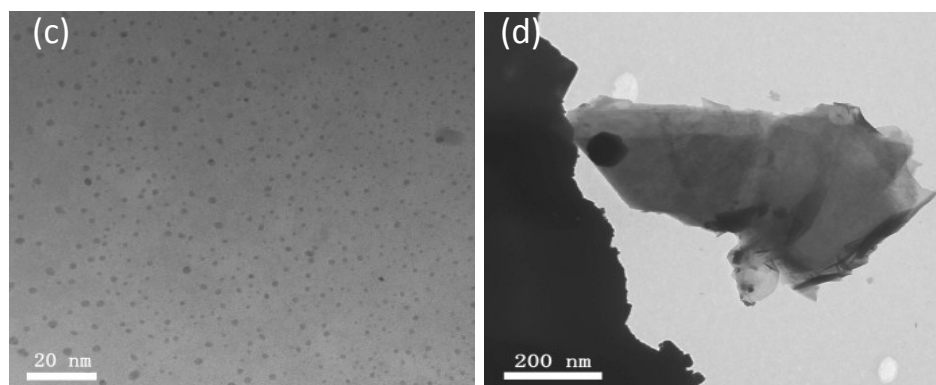
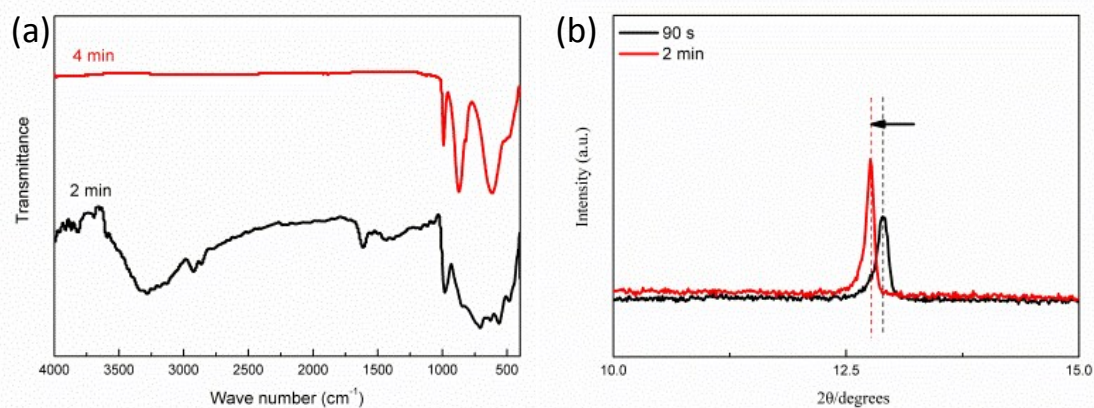


Fig. S6 (a) The IR spectra of the products left after thermal exfoliation under 530°C for different time. After 4 min, the organics had been completely removed. (b) The XRD patterns of the products left after thermal exfoliation under 600°C for different time. After 90 s, the powder had fully turn to α -MoO₃. With extension processing time to 2 min, the crystal structure became more well-ordered as XRD peak became more sharp and slightly moved to smaller angle. (c) The TEM image of MoO₃ QDs obtained under 530°C for 4 min. (d) The TEM image of MoO₃ nanosheets obtained with prolonged thermal exfoliation time.

6. MoO₃ QDs with irregular shapes

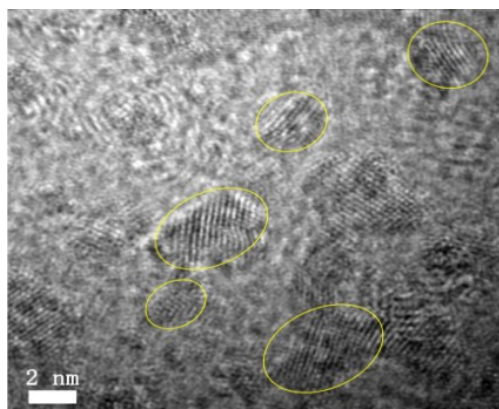


Fig. S7 The HRTEM image of MoO₃ QDs with irregular shapes.