Electronic Supplementary Information

A general one-step approach for in situ decoration of MoS₂ nanosheets with inorganic nanoparticles

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Experimental Section

Synthesis of noble metal nanoparticles decorated MoS₂ nanosheets (MoS₂-Pt and MoS₂-Au). Typically, MoS₂-COOH (20 mg) was dispersed in DEG (30 mL) and the mixture was treated with an ultrasonic bath (40 kHz) for 15 min. Then, K₂PtCl₄ (20.8 mg, 0.05 mmol) or HAuCl₄·3H₂O (19.7 mg, 0.05 mmol) was added and stirred for 30 min. The above mixture was heated to 200 °C for 20 min under the protection of argon flow and constant stirring. 7.5 mg of NaBH₄ in 10 mL of DEG was injected rapidly into the above hot mixture. The resulting mixture was further heated at 200 °C for 1 h. The final product of MoS₂-Au or MoS₂-Pt was separated by centrifugation and washed with water.

For comparison, MoS₂-Pt was also synthesized by using sodium citrate instead of NaBH₄ as reducing agent. In addition, graphene was also employed as substrate to

fabricate graphene-Pt nanohybrid with the same synthetic procedure as MoS₂-Pt.

Synthesis of Fe_3O_4 nanoparticles decorated MoS_2 nanosheets ($MoS_2-Fe_3O_4$). Typically, NaOH (100 mg) was added into DEG (10 mL), heated at 120 °C for 1 h in a argon atmosphere, and cooled down to 70 °C to produce a NaOH/DEG stock solution. MoS_2 -COOH (20 mg) was dispersed in DEG (30 mL) and the mixture was treated with an ultrasonic bath (40 kHz) for 15 min. Then, FeCl₃ (80 mg, 0.49 mmol) was added and stirred for 30 min. The above mixture was heated to 200 °C for 20 min under the protection of argon flow and constant stirring. 3.5 mL of 70 °C NaOH/DEG stock solution was injected rapidly into the above hot mixture. The resulting mixture was further heated at 200 °C for 1 h. The final product of MoS_2 - Fe_3O_4 was separated by centrifugation and washed with water.

Synthesis of semiconductor nanoparticles decorated MoS₂ nanosheets (MoS₂-CdS and MoS₂-PbS). Typically, MoS₂-COOH (20 mg) was dispersed in DEG (30 mL) and the mixture was treated with an ultrasonic bath (40 kHz) for 15 min. Then, CdCl₂ (18.4 mg, 0.1 mmol) or Pb(CH₃COO)₂·3H₂O (40 mg, 0.1 mmol) was added and stirred for 30 min. The above mixture was heated to 200 °C for 20 min under the protection of argon flow and constant stirring. 25 mg of Na₂S·9H₂O in 10 mL of DEG solution was injected rapidly into the above hot mixture. The resulting mixture was further heated at 200 °C for 1 h. The final product of MoS₂-CdS and MoS₂-PbS was separated by centrifugation and washed with water.





Fig. S1. (a) Representative AFM image of MoS_2 -COOH nanosheets. (b) Height profile of corresponding planes along the lines in (a).

Fig.	S2
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Fig. S2. (a) FTIR spectra of MPA, MoS₂-COOH and unmodified MoS₂ nanosheets. XPS survey spectrum (b) and C1s spectrum (c) of MoS₂-COOH. (d) TGA curves of MoS₂-COOH and unmodified MoS₂ nanosheets.

The successful introduction of carboxylic groups was elucidated by FTIR and XPS. For comparison, we also synthesized MoS₂ nanosheets using the same procedure as MoS₂-COOH except the addition of mercaptopropionic acid (MPA) ligand and this sample was designated as unmodified MoS₂. As can be seen in Fig. S2a, the MPA sample shows a strong absorption band at 2574 cm⁻¹ corresponding to the –SH stretching. After conjugation with MoS₂, this band disappeared in the resulted MoS₂-COOH sample, possibly caused by the interaction between –SH with MoS₂. The exfoliation process leads to deformation of MoS₂'s crystal structure and

formation of numerous internal edges and defects. On the other hand, previous works suggested that the sites of internal edges and defects possess high affinities toward –SH group.^{1,2} In addition, the MoS₂-COOH sample shows obvious absorption bands at 2980, 2870 and 1457 cm⁻¹ due to the C-H stretching, 1101 cm⁻¹ corresponding to C-O stretching, and 1044 cm⁻¹ associated with C-S stretching, demonstrated the successful conjugation of MPA ligand.

According to the full survey spectrum (Fig. S2b), the elements of Mo, S, C and O are found for the MoS₂-COOH sample. The mass percent of C and O were calculated according to the XPS results and found that the MoS₂-COOH sample shows much higher content of C (2.6 wt% and 0.9 wt% for MoS₂-COOH and unmodified MoS₂, respectively) and O (1.9-2.5 wt% and 0.2 wt% for MoS₂-COOH and unmodified MoS₂, respectively) than that of unmodified MoS₂ nanosheets, further confirmed the successful attachment of carboxylic groups. In addition, the high-resolution C1s spectrum indicates the presence of O=C-O and C-C bonds for the MoS₂-COOH was determined by TGA as shown in Figure S2d. The unmodified MoS₂ sample shows very low weight loss (about 1.3 wt%) between 150-700 °C, presumably due to the presence of residual organic component resulted from *n*-BuLi. In contrast, the MoS₂-COOH sample shows much higher weight loss between 150-700 °C (7.1 wt%), further confirming that the carboxylic groups have been successfully introduced onto the surface of MoS₂ nanosheets.

Reference

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Fig. S3. Representative TEM images of MoS₂-Pt (a), MoS₂-Au (b), MoS₂-Fe₃O₄ (c), MoS₂-CdS (d) and MoS₂-PbS (e). (f) Magnetization curve (at 300 K) of MoS₂-Fe₃O₄.

Fig. S4



Fig. S4. Photographs of MoS_2 -Fe₃O₄/PVA nanocomposite film.

Fig. S5



Fig. S5. Representative TEM images of MoS_2 -Pt (a,b) and MoS_2 -CdS (c,d) by using unmodified MoS_2 as a support.





Fig. S6. TEM images of graphene-Pt.





Fig. S7. TEM images of MoS_2 -Pt by using sodium citrate as reducing agent.

Fig.	S8



Fig. S8. Representative TEM images of MoS_2 -Fe₃O₄ with R_{wt} (FeCl₃/MoS₂-COOH) value of 1/1 (a) and 4/1 (b). (c) EDS spectrum of MoS_2 -Fe₃O₄ shown in (a). (d) Magnetization curves (at 300 K) of MoS_2 -Fe₃O₄ with R_{wt} (FeCl₃/MoS₂-COOH) value of 1/1 and 4/1.





Fig. S9. Representative TEM images of MoS₂-Fe₃O₄/Pt sample.





Fig. S10. (a) Concentration change in 4-nitrophenol compounds (C_t/C_0) in the presence of MoS₂-Fe₃O₄/Pt nanocatalysts. (b) Plot of ln (C_t/C_0) against time for the reduction of 4-nitrophenol with MoS₂-Fe₃O₄/Pt nanocatalysts.