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A novel route to the synthesis of bulk and well dispersed alumina-supported Ni_2Mo_3N catalysts via single-step hydrogen thermal treatment

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Supplementary Information

Synthesis of alumina supported Ni_2M_3N by the TPN method

The preparation of Ni_2Mo_3N/γ-Al_2O_3 by the conventional temperature-programmed reduction method in NH_3 flow is described as follows:

The γ-Al_2O_3 support was impregnated simultaneously with an aqueous solution containing $Ni(CH_3COO)_2{\cdot}6H_2O$ and $(NH_4)_6Mo_7O_{24}{\cdot}2H_2O$ with a molar ratio of 14:3 in 15 wt % $NH_3{\cdot}H_2O$ solution with stirring for 3 h. The mixture was then filtered. The solid was dried at 393 K for 3 h, and then calcined in air at 773 K for 5 h to obtain the NiO-MoO_3/γ-Al_2O_3 precursor for nitrides. The supported oxides precursor was then heated in a NH_3 flow at a mass rate of 6500 h^{-1}. The temperature was increased linearly at a rate of 6 K/min from room temperature to 623 K, and then at a rate of 1 K/min to 923 K, finally kept at this temperature for 2 h. The product was naturally cooled to room temperature and passivated for 7 h in a flow of 1% (v/v) O_2/N_2 to achieve 23 wt % Ni_2Mo_3N/γ-Al_2O_3.

Table S1 The chemical composition of bulk Ni_2Mo_3N prepared by the hydrogen thermal method

	Ni content (wt %)	Mo content (wt %)	N content (wt %)	Elemental composition
Bulk Ni_2Mo_3N	26.92	66.85	2.93	$Ni_2Mo_{3.04}N_{0.91}$
Theoretical value	28.00	68.66	3.34	Ni_2Mo_3N

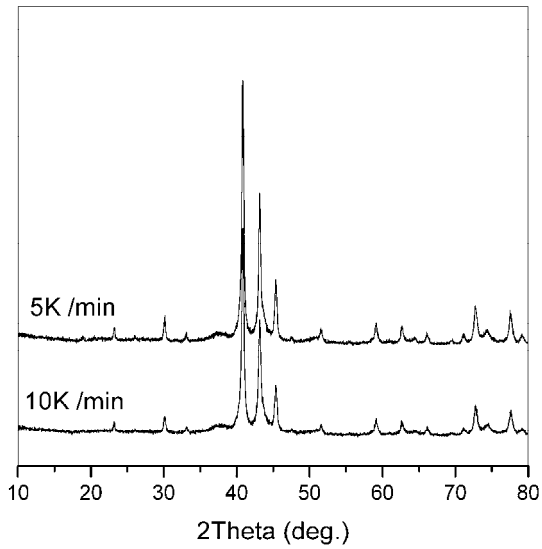

Fig. S1. Bulk Ni_2Mo_3N prepared with different ramp rates by the hydrogen thermal method

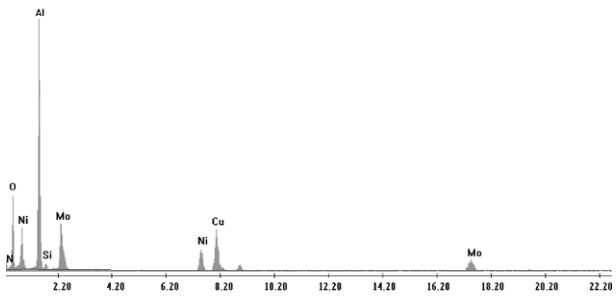

Fig. S2. EDX pattern of Ni_2Mo_3N/Al_2O_3 prepared by the hydrogen thermal method

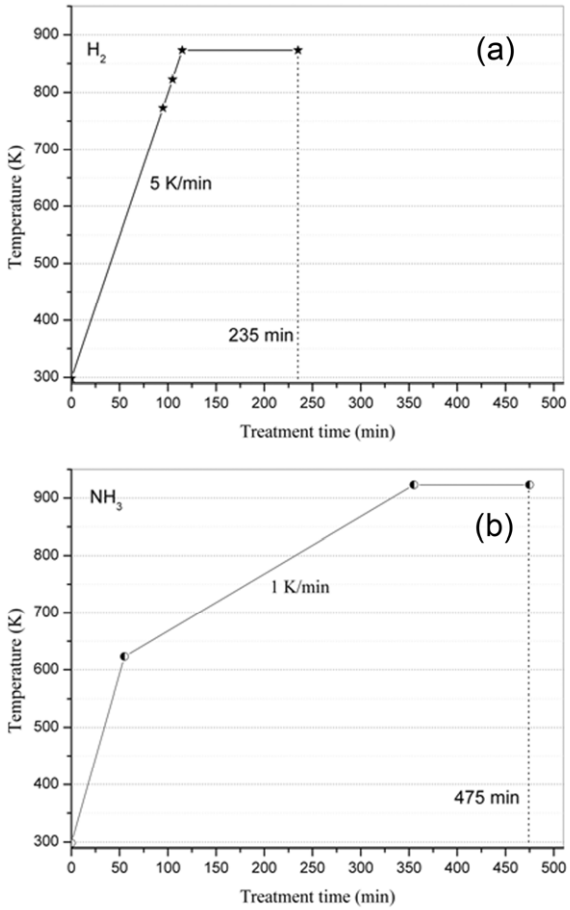

Fig. S3 The synthesis processes of 23 wt % Ni_2Mo_3N supported on alumina prepared (a) by hydrogen thermal method and (b) by the conventional TPN method.