Supporting Information

Molybdenum Modified Nickel Phyllosilicate as a High Performance Bifunctional Catalyst for Deoxygenation of Methyl Palmitate to Alkanes under Mild Conditions

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Characterizations:

H_2-TPR and NH_3-TPD experiment

H_2 temperature programmed reduction (TPR) and NH_3 temperature programmed desorption (TPD) were both carried on a home-built reactor system with a thermal conductivity detector (TCD).

For H_2-TPR tests, 50 mg of Ni based catalysts were added into the quartz tube and heated to 500 ^oC under Ar flow at rate of 10 ^oC/min and kept for 1 hour to remove the impurities. Then the treated catalysts were cooled down to 100 ^oC. After removing the impurities, the samples were heated to 850^oC under H_2 flow at rate of 10 ^oC/min. The ice-salt-bath was employed to remove the moisture from the hot stream. The dried stream was analyzed by the TCD.

For NH_3-TPD tests, 90 mg of reduced Ni catalysts was heated to 300 ^oC to remove the impurities as pretreatment. This pretreatment kept for 1 hour. After cooling down, the sample was saturated with pure NH_3 for 1 hour. Subsequently, the physisorbed NH_3 was removed under Ar flow at 100 ^oC for 0.5 hour. After that, the sample was heated to 700 ^oC in a rate of 10 ^oC/min. The released stream was analyzed by the TCD.

XRD experiment

XRD tests were conducted on X-ray diffractometer (TTR-III, Rigaku Corp., Japan) with Cu Kα radiation.

TEM and HRTEM

TEM and HRTEM measurements were both carried out on a JEM 2011 electron microscope.

XPS experiment

XPS measurements were obtained on an X-ray photoelectron spectrometer (ESCALAB250, Thermo-VG Scientific, USA) with monochromatized Al Kα radiation.

FT-IR experiment

FT-IR spectra were recorded by a Nicolet 8700 FT-IR spectrometer.

N_2 adsorption-desorption isotherms

N_2 adsorption-desorption isotherms measurements were carried out on Micromeritics Tristar II 3020M instrument via N_2 adsorption at -195.8°C.

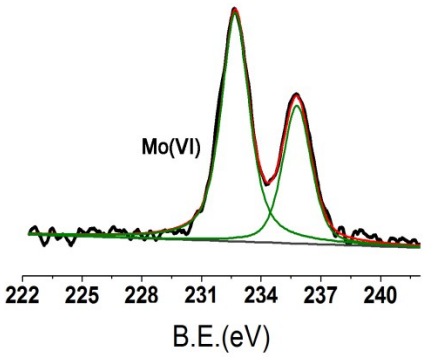


Figure S1. Mo 3d XPS spectra of as-calcined 3%Mo-Ni@PSi(B) precursors.

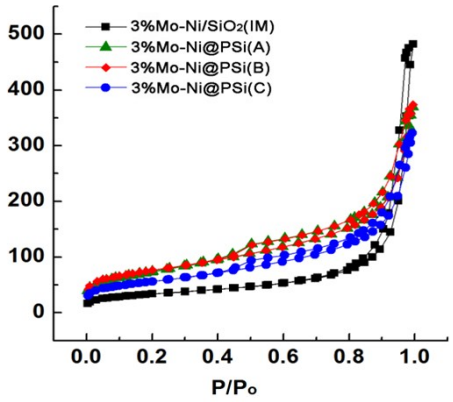


Figure S2. N_2 adsorption-desorption isotherms of reduced Mo doping Ni catalysts.

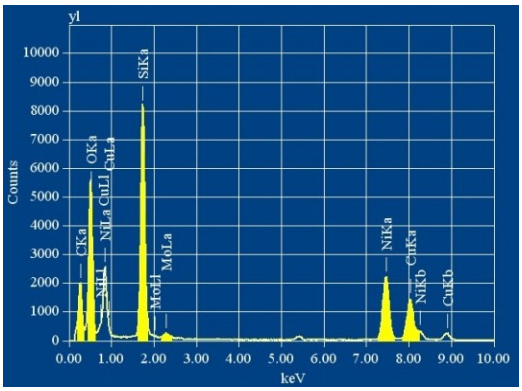


Figure S3. The EDS imagae for the 3%Mo-Ni@PSi(B) catalyst

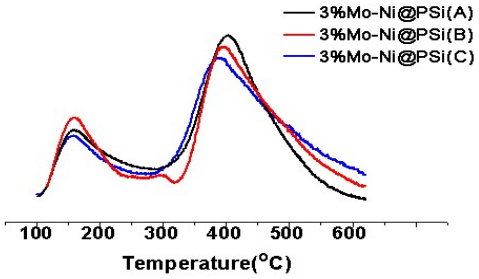


Figure S4. The NH_3-TPD profiles of 3%Mo-Ni@PSi catalysts

Table S1. Comparison Ni species proportion over the surfaces of fresh and used 3%Mo-Ni@PSi(B) catalysts

Sample	Ni(0) Ratio(%)	NiO Ratio(%)	1:1 Ni-PSi Ratio(%)	2:1 Ni-PSi Ratio(%)
3%Mo-Ni@PSi(B)-fresh	8.7	6.6	66.7	18.0
3%Mo-Ni@PSi(B)-used	2.5	10.9	68.7	17.9

Table S2. Comparison Mo species proportion over the surfaces in 3%Mo-Ni@PSi(B)

Catalyst	Mo(IV) Ratio (%)	Mo(V) Ratio (%)	Mo(VI) Ratio (%)
3%Mo-Ni@PSi(B)-precursor	--	--	100
3%Mo-Ni@PSi(B)-fresh	6.4	33.9	59.6
3%Mo-Ni@PSi(B)-used	5.7	20.3	74.0

Table S3. Catalytic performances of 3%Mo-Ni@PSi(B) catalysts from three different batches

Preparation date	Conv.(%)	Yield(%)	
		C_{15}	C_{16}
2016.03	100	69.2	30.4
2016.06	100	70.0	28.3
2017.07	100	72.9	27.0

The reproductibility of the method was tested in 3%Mo-Ni@PSi(B) catalysts from three different batches .The performances were basically consistent, reflecting the preparation method was reproductibility. However, it should be noticed that the oxidation would gradually cause the inactivation of this superior 3%Mo-Ni@PSi(B) catalyst.

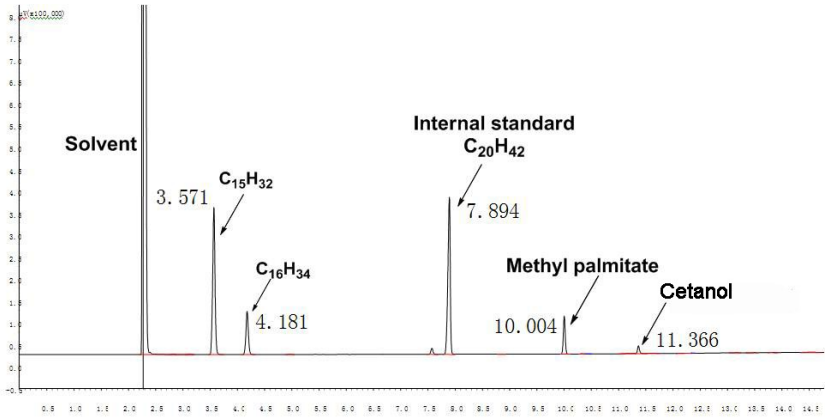
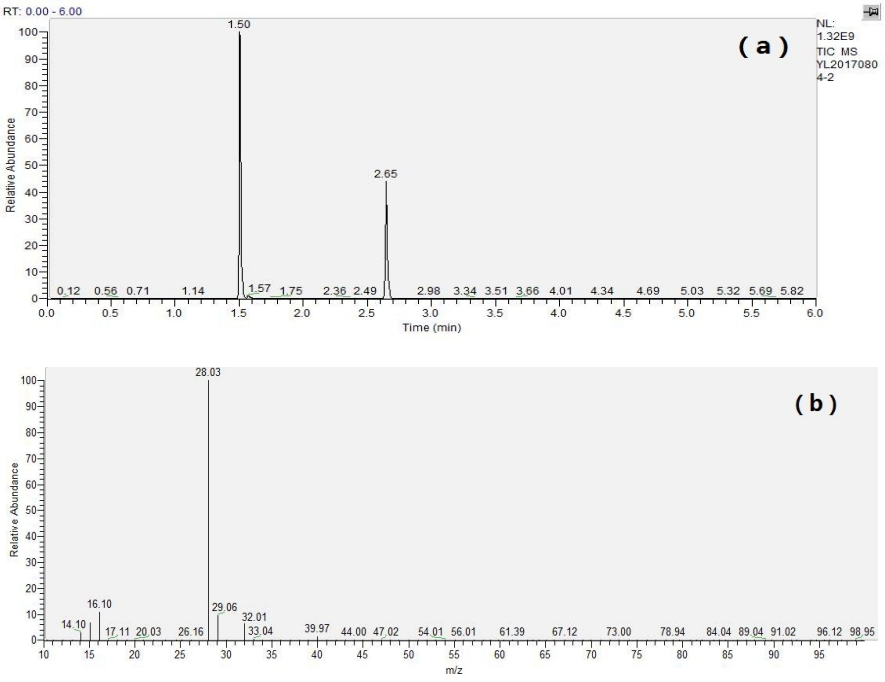


Figure S5. The GC traces of the reaction mixtures. (Eicosane was employed as internal standard for the quantitative analysis. The remain times of substrate, intermediate and products were labeled in Figure S5.)



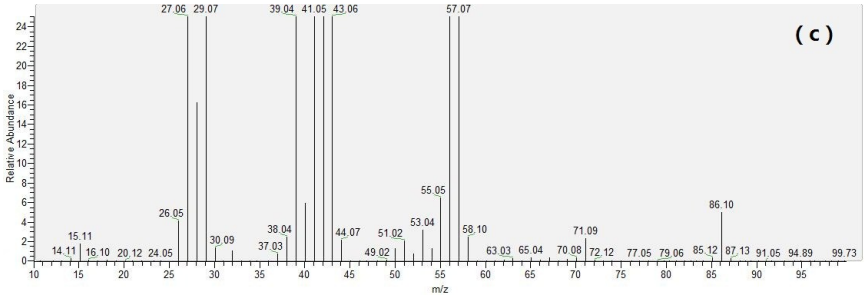


Figure S6. GC-MS detection for the gas product yielded in the model reaction: (a) the GC traces of gas product; (b) MS for the peak at 1.5 min (CO product); (c) MS for the peak at 2.65 min (C_6H_{14} solvent).