Supporting Information

Heterogeneous Nb-containing catalyst/N,N-dimethylacetamide-salts mixtures: Novel and efficient catalytic systems for dehydration of fructose

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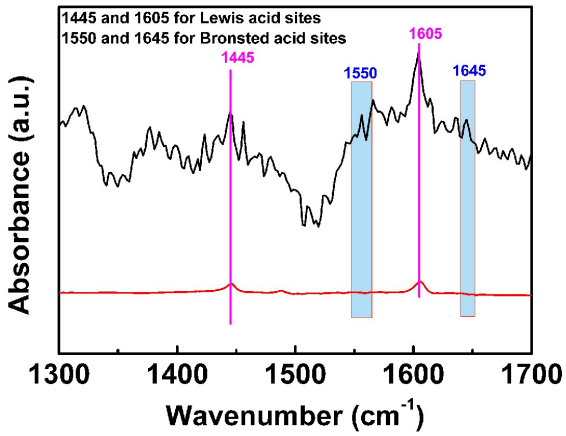

Fig. S1. Pyridine absorption FT-IR analysis of Nb-NTMPA and Nb_2O_5, which were determined by pyridine FT-IR on the Nicolet NEXUS 670 FT-IR spectrometer with a resolution of 2 cm^{-1}. The samples were finely ground and pressed into a self-supporting wafer (diameter 15 mm, 42 mg). The wafers were evacuated in the IR cell at 150 °C for 1 h under a vacuum in order to remove physisorbed water. After the temperature decreased to room temperature, IR spectra were recorded in the range from 2500 to 1000 cm^{-1}. Subsequently, the samples were followed by the adsorption of purified pyridine vapor at room temperature for 50 min. The pyridine absorption IR spectra were recorded after subsequent evacuation of the infrared cell at 200 °C. The spectra presented were obtained by subtracting the spectra recorded before and after pyridine adsorption. The quantity of Lewis and Brønsted acid sites was evaluated according to the equation in the literature (*J. Catal., 1993, 141, 347-354*):

$$C(\text{pyridine on Lewis sites}) = 1.42 I_A(L) R^2 / W$$

$$C(\text{pyridine on Brønsted sites}) = 1.88 I_A(B) R^2 / W$$

In the equation, C=concentration (mmol/g catalyst); I_A(L or B)=integrated absorbance of Lewis or Brønsted acid band (cm^{-1}); R=radius of catalyst disk (cm); W=weight of disk (mg).

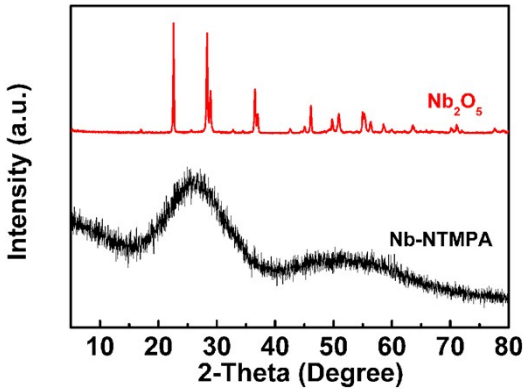

Fig. S2. XRD patterns of Nb-NTMPA and Nb_2O_5.

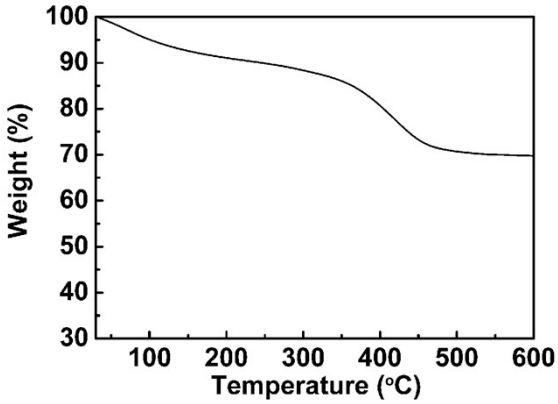

Fig. S3. The thermogravimetric analysis of Nb-NTMPA. The weight loss (about 5%) up to 150 °C is attributable to the loss of intercalated and adsorbed water and ethanol molecules in the sample. The obvious weight loss (about 20%) occurred between 350 °C and 450 °C corresponds to the burning of organic fragments of the hybrid framework.

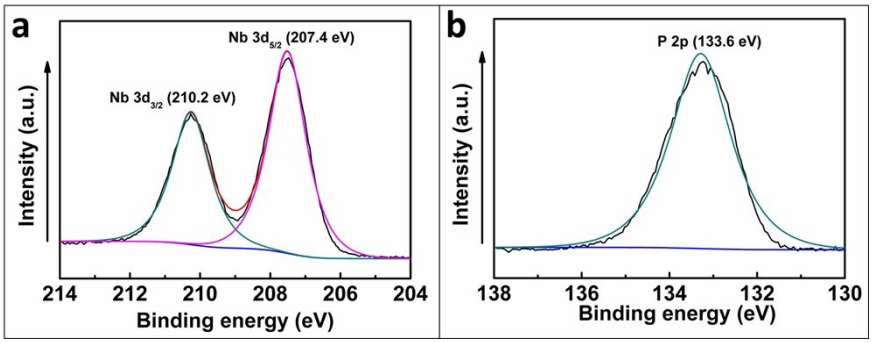

Fig. S4. XPS spectra of Nb 3d (a) and P 2p (b) for the recovered Nb-NTMPA after reused three times.

Table S1. *n*-Butylamine titration method using various Hammett indicators for the acid strength and amounts, which was similar with the reported route (C*arbohydr. Res., 2013, 368, 78*).[a]

Sample	Weak and medium strong acid ($-3.0 \le H_0 \le 6.8$)	Strong acid ($-8.2 \le H_0 \le -3.0$)	Very strong acid ($H_0 \le -8.2$)	Total acid
Nb-NTMPA	0.18 mmol/g	0.06 mmol/g	0	0.24 mmol/g
Nb_2O_5	0.07 mmol/g	0	0	0.07 mmol/g

[a]The Hammett indicators included anthraquinote (pKa=-8.2), chalcone (pKa=-5.6), dicinnamalacetone (pKa=-3.0), methyl yellow (pKa=3.3), and neutral red (pKa=6.8). Before the examination, 0.1 g samples were pretreated at 120 ^oC for 12 h in the atmosphere of He.

Table S2. Properties of the prepared Nb-NTMPA.

Entry	Sample[a]	BET surface area (m^2/g)[b]	Pore volume (cm^3/g)[c]	Pore diameter (nm)[d]
1	Nb-NTMPA	25.2	0.14	5.5
2[e]	Nb-NTMPA	20.7	0.12	4.7

[a]The samples were degassed at 100 °C for 24 h. [b]Surface area based on multipoint BET method. [c]Pore volume based on BJH method. [d]Pore diameter based on BJH method. [e]The Nb-TPMA was the recovered one after reused for three times.

Table S3. The contents of various elements on the surface of Nb-NTMPA determined by XPS examination.

Element	Content of various elements on the surface (Atomic%)[a]	
	Fresh Nb-NTMPA	Spent Nb-NTMPA
Nb	4.51	4.35
N	6.24	5.97
P	9.31	9.72
O	37.27	39.05
C	42.67	40.91