

Supporting Information

RSC Advances

Niobium Phosphotungstates: Excellent Solid Acid Catalysts for the Dehydration of Fructose to 5-Hydroxymethylfurfural under Mild Conditions

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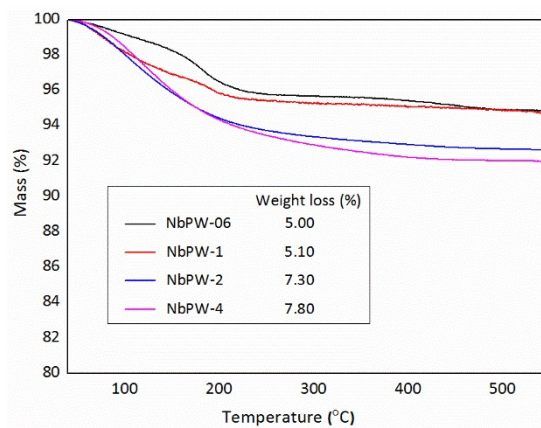


Fig. S1 TG analysis of the NbPW material.

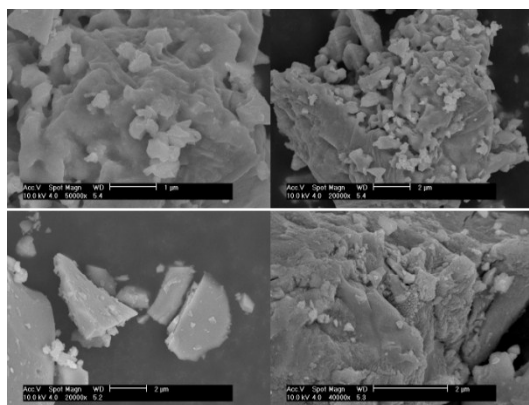


Fig. S2 SEM images of NbPW-06.

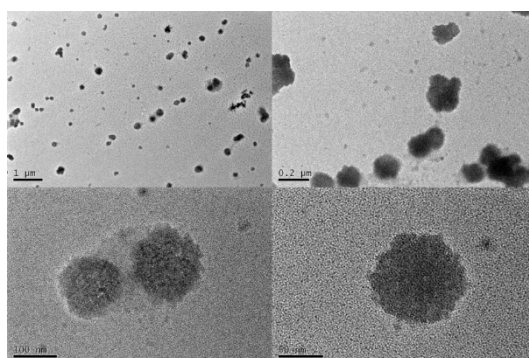


Fig. S3 TEM images of NbPW-06.

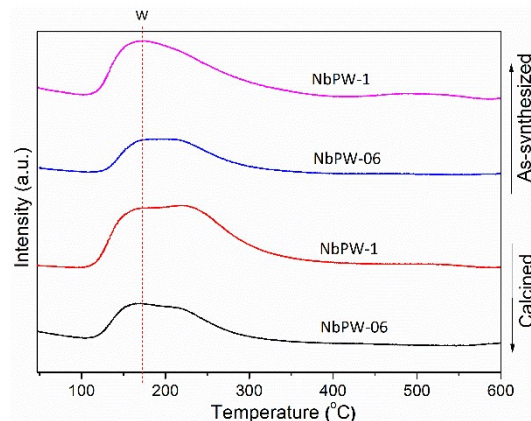


Fig. S4 NH₃-TPD spectra of NbPW-06 and NbPW-1. Desorption peaks characteristic of weakly acidic (W) sites are indicated.

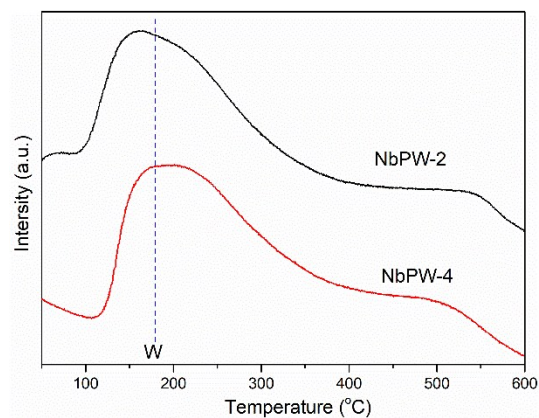


Fig. S5 NH₃-TPD spectra of NbPW-2 and NbPW-4. Desorption peaks characteristic of weakly acidic (W) sites are indicated.

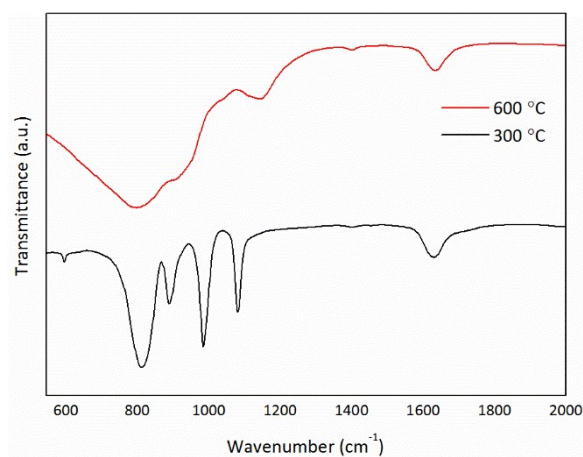


Fig. S6 FTIR spectra of NbPW-06 calcined at 300 °C and 600 °C.

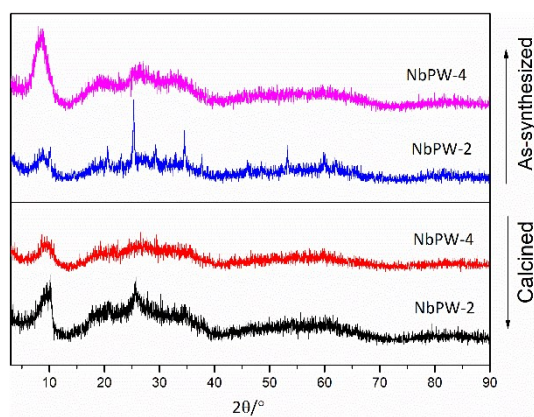


Fig. S7 The X-ray diffraction patterns of NbPW-2, and NbPW-4.

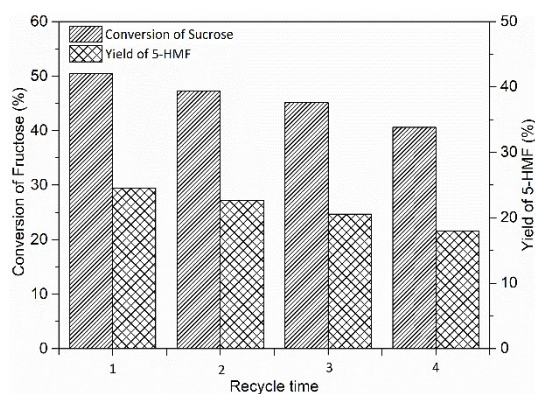


Fig. S8 Reusability of NbPW-06 catalyst (conditions: $[\text{fructose}]_0 = 56 \text{ mM}$, NbPW-06 = 50 mg, 5 ml DMSO, 80 °C, 5 min).

Table S1 Catalytic conversion of fructose to 5-HMF by NbPW.
 ($[\text{fructose}]_0 = 56 \text{ mM}$; catalyst=50 mg; 5 ml DMSO; T= 80 °C)

Entry	Catalyst	t (min)	Fructose Conv. (%)	5-HMF (%)	Selectivity (%)	TOF (min^{-1})
1	NbPW-06	6	55.4	29.8	53.9	0.313
2	NbPW-1	6	59.4	23.6	39.8	0.298
3	NbPW-2	6	58.6	17.2	29.3	0.267
4	NbPW-4	6	46.3	8.7	18.7	0.168

TOF = turnover frequency ($\text{mol 5-HMF}_{\text{produced}}$ per mol acid sites per minute at 6 min).

Table S2 Characterization of the recycled NbPW-06 by NH_3 -TPD and TGA analysis.

Recycle number	Total acid sites (mmol g^{-1})	Weight loss (%)	Carbon deposition (%)
1	0.89	5.21	-
2	0.88	5.29	0.07
3	0.85	5.33	0.12
4	0.81	5.40	0.19

Table S3 The BET characterization of the NbPW catalysts.

Catalyst	BET surface area (m ² /g)
HPW	10.3
NbPW-06	39.9
NbPW-1	37.5
NbPW-2	37.1
NbPW-4	36.4

1.1 BET characterization

The BET surface area of the NbPW catalyst increased slightly compared with pure HPW, resulting into the uniform distribution of protons on the surface of the catalyst (Table S3). Therefore, more acid sites could be expected on the surface, which is consistent with the excellent catalytic activity.

1.2 Reusability of NbPW-06 catalyst

The slight deactivation processes of the catalyst can be observed from Figure S8. The results showed the slight decreases in the yield of 5-HMF and the conversion of fructose, which can be attributed to the minor decrease in the activity of the NbPW materials or partial humins formation during the later reaction cycles.