To: Catalysts

Supporting Information

Phosphate enrichment of niobium based catalytic surfaces in relation to reactions of carbohydrate biomass conversion: the case studies of inulin hydrolysis and fructose dehydration

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Figures and Tables

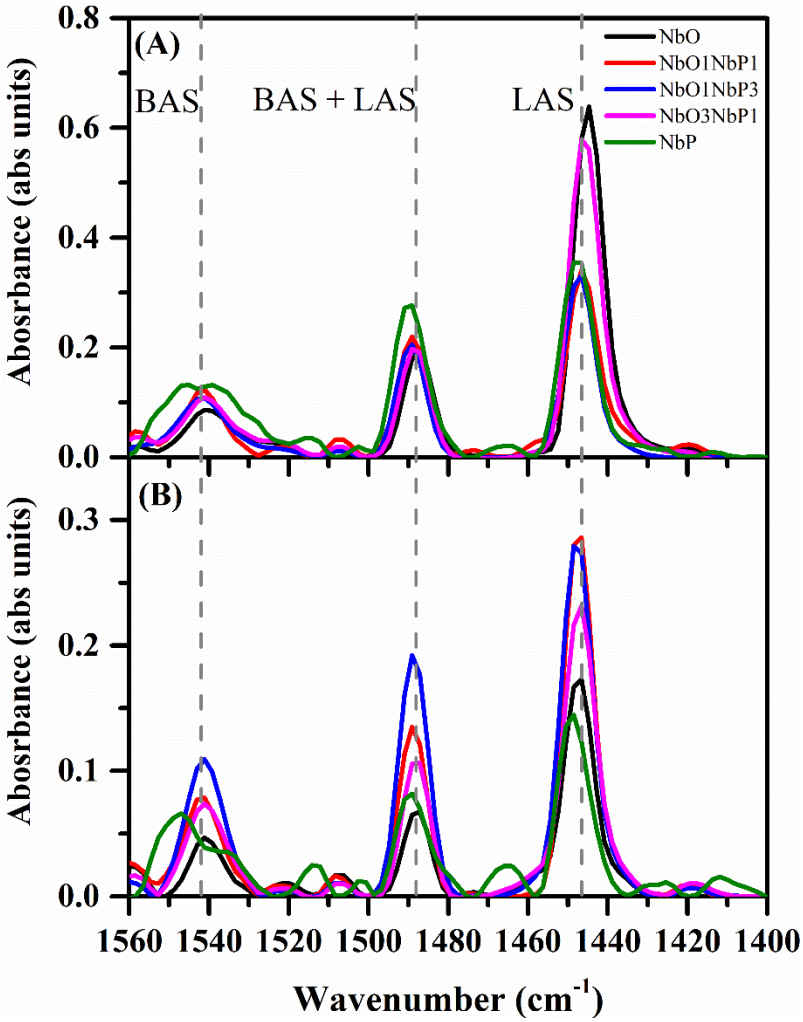

Figure S1 FT-IR spectra of catalyst samples with pyridine vapor (A) and pyridine aqueous (B) after pyridine desorption at 150°C. The baselines of all spectra have been corrected.

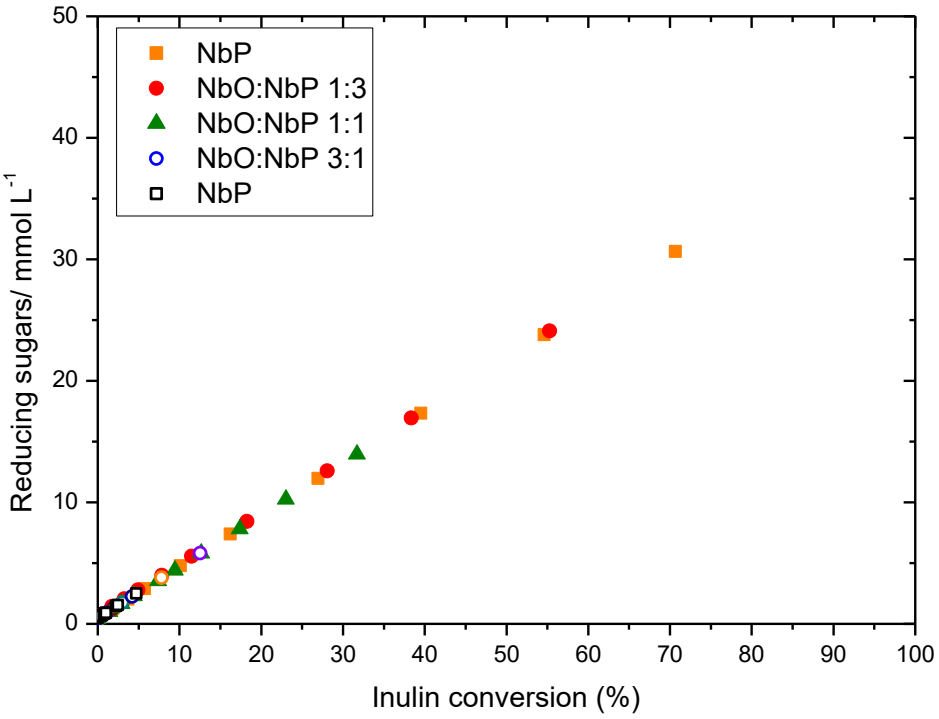

Figure S2 Cumulative trend of the reducing sugar concentrations against inulin conversion, taken as an index of reaction progress.

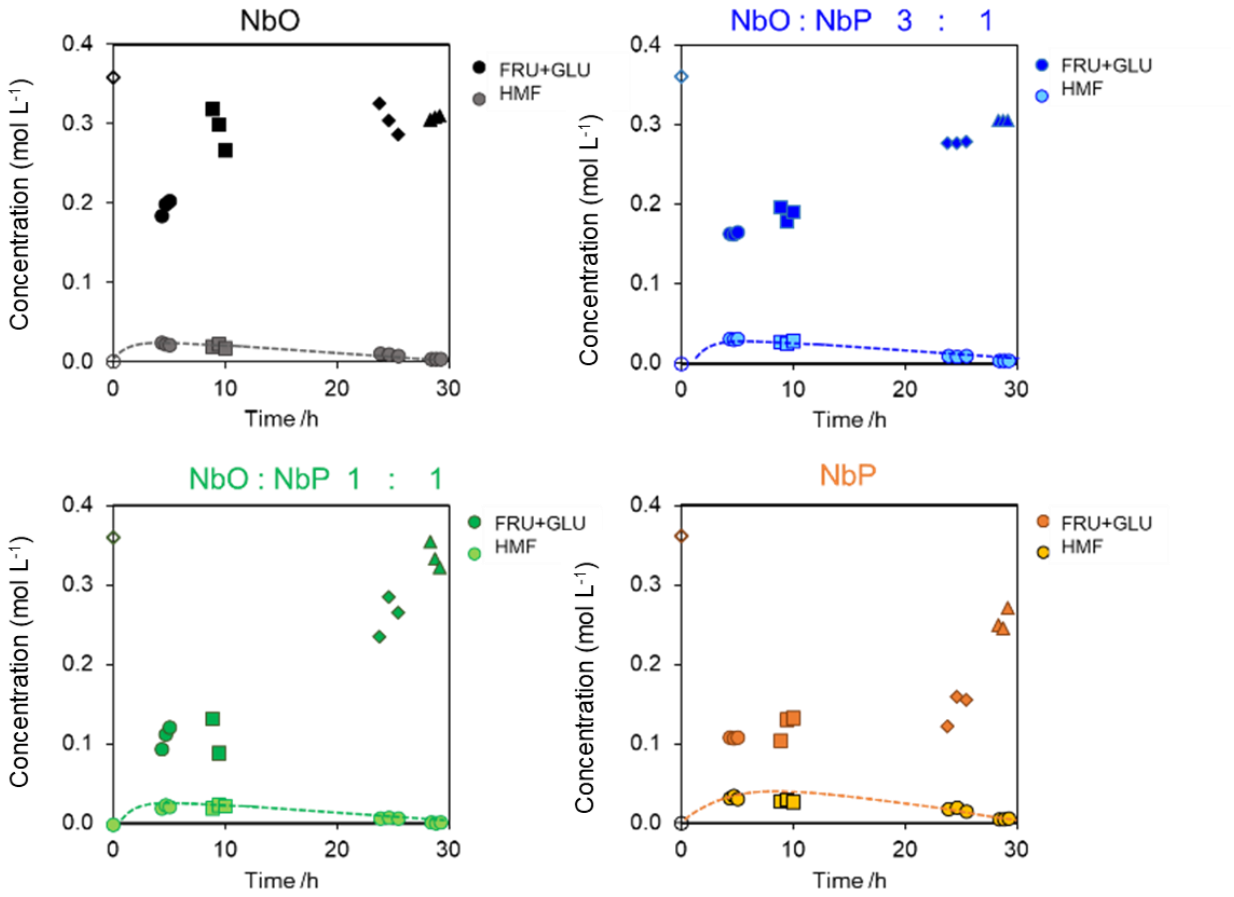

Figure S3 Concentration profiles obtained from the continuous-flow reaction of glucose/fructose dehydration collected at 120°C: concentration of reagents (GLU+FRU) and formed HMF as a function of reaction time at different contact times: ● = 6 min· g^{-1}·mL^{-1}; ■ = 10 min· g^{-1}·mL^{-1}; ◊ = 15 min· g^{-1}·mL^{-1};▲ = 7.5 min· g^{-1}·mL^{-1}.

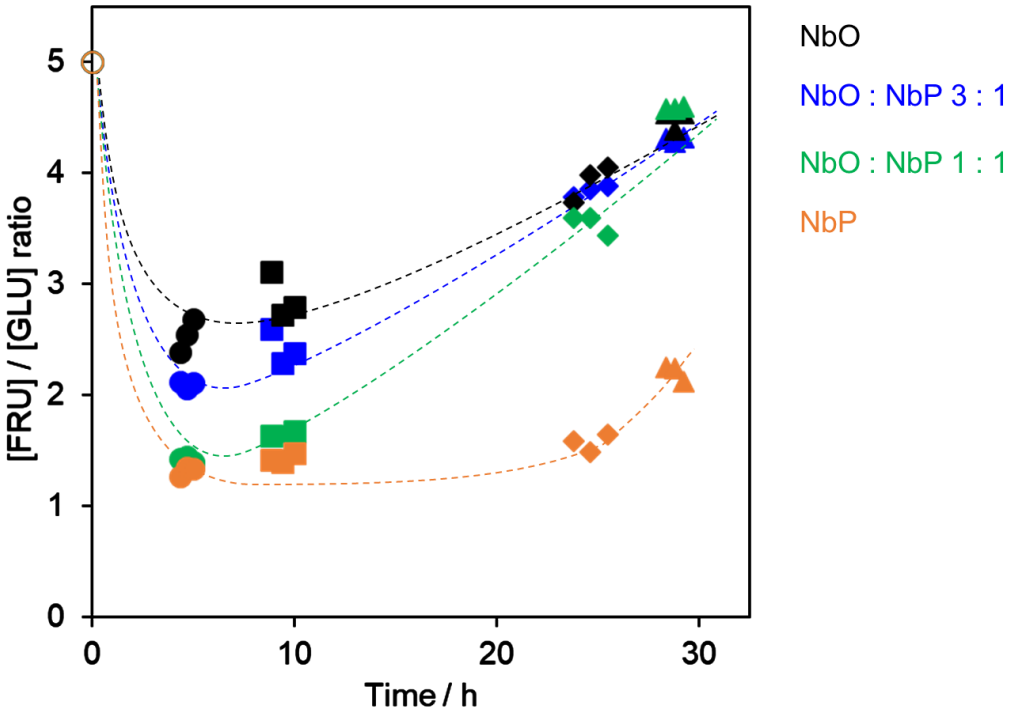

Figure S4 Plot of fructose to glucose ratio as a function of reaction time in the continuous-flow reaction of glucose/fructose dehydration at 120°C at different contact times: ● = 6 min g^{-1} mL^{-1}; ■ = 10 min g^{-1} mL^{-1}; ◊ = 15 min g^{-1} mL^{-1};▲ = 7.5 min g^{-1} mL^{-1}.

Table S1 EDX and XPS compositions of Nb-based samples

	EDX composition (at.%)						*XPS surface composition (at.%)*			
	Areal analysis			*Punctual analysis*						
	Nb	**P**	**O**	**Nb**	**P**	**O**	**Nb**	**P**	**O**	**C**
NbO	26.6±0.6 (67.8 wt.%)	-- (-- wt.%)	73.4±0.8 (32.2 wt.%)	27.4±0.5 (68.6 wt.%)	-- (.. wt.%)	72.6±0.8 (31.3 wt.%)				
NbO:NbP 3:1	21.9±0.5 (60.8 wt.%)	4.1±0.1 (3.8 wt.%)	74.0±0.77 (35.4 wt.%)	20.7±0.8 (58.7 wt.%)	5.6±0.1 (5.3 wt.%)	73.8±0.8 (36.1 wt.%)	18.8	4.3	58.6	18.3
NbO:NbP 1:1[a]	18.6±0.5 (54.9 wt.%)	7.0±0.1 (6.9 wt.%)	74.0±0.8 (37.7 wt.%)	15.9±0.5 (49.8 wt.%)	8.9±0.1 (9.2 wt.%)	74.6±0.8 (40.1 wt.%)	15.0	6.6	50.0	28.4
NbO:NbP 1:3[a]	16.2 ±0.5 (50.1 wt.%)	9.4±0.1 (9.7 wt.%)	73.8±0.8 (39.4 wt.%)	14.8±0.5 (47.2 wt.%)	10.2±0.1 (10.9 wt.%)	74.4±0.8 (41.0 wt.%)	14.5	9.2	58.6	17.6
NbP[a]	14.1±0.3 (45.7 wt.%)	10.8±0.1 (11.7 wt.%)	74.4±0.6 (41.6 wt.%)	14.5±0.5 (46.5 wt.%)	11.0±0.2 (11.8 wt.%)	73.8±0.8 (40.8 wt.%)	19.2	9.3	52.4	19.0

[a] Potassium (K) was detected as impurity (< 1 wt.%)

Table S2 Nature and surface density of the acid sites in NbO and NbP solids and in physical mixtures, determined by FT-IR with pyridine (PY) adsorption in vapor phase with/without water and by solid-liquid phase titrations with phenyl-ethylamine PEA.

	Nature of the acid sites		Acid site density[c]	
	$Py_{(vap)}$[a]	$Py_{(aq)}$[b]	Intrinsic acidity	Effective acidity
Catalyst	LAS/BAS ratio		Total acid sites (μequiv m^{-2})	
NbO	3.63	2.86	3.23 (73%)[d]	2.16 (60%)[d]
NbO:NbP(3:1)	2.67	2.05	3.70 (66%)	2.46 (55%)
NbO:NbP(1:1)	1.85	2.18	3.62 (66%)	2.62 (61%)
NbO:NbP(1:3)	1.48	1.69	4.07 (70%)	2.99 (59%)
NbP	0.87	0.91	3.32 (72%)	3.31 (62%)

[a] Pyridine contacted in vapor phase, quantification after desorption at 150°C;

[b] Pyridine contacted in aqueous solution, quantification after desorption at 150°C;

[c] Adsorption temperature, 30°C; solvent, cyclohexane for intrinsic acidity and water/isopropanol mixture for effective acidity measurements;

[d] Percent of strong acid sites.