

Electronic Supplementary Information for
**Glucose production from saccharides using layered transition metal oxide and
exfoliated nanosheets as a water-tolerant solid acid catalyst**

Atsushi Takagaki, Caio Tagusagawa, and Kazunari Domen*

*Department of Chemical System Engineering, School of Engineering, The University of Tokyo,
7-3-1 Hongo, Bunkyo-ku, Tokyo 113-8656, Japan*

Content

Experimental

Table S1: Hydrolysis of polysaccharides (starch and cellulose) over HNbMoO_6

**Figure S1: Time courses of cellobiose hydrolysis over layered HNbMoO_6 , Amberlyst-15
and Nafion NR50**

Figure S2: FT-IR spectrum for glucose-intercalated HNbMoO_6

Experimental:

Catalyst Preparation

LiNbMoO_6 was obtained by calcination of a stoichiometric mixture of Li_2CO_3 , Nb_2O_5 , and MoO_3 at 853 K for 24 h with one intermediate grinding. The proton-exchange reaction was carried out by shaking the Li form in 1 M HNO_3 at room temperature for 1 week.

HTiNbO_5 nanosheets were prepared by exfoliation – aggregation of layered HTiNbO_5 . Exfoliated TiNbO_5^- sheets were obtained by adding 15 wt% tetra(*n*-butylammonium)hydroxide (TBA^+OH^-) solution to 150 mL of distilled water containing 2.0 g of layered HTiNbO_5 . The suspension was then centrifuged, and the supernatant solution was collected. The addition of dilute HNO_3 aqueous solution to 30 mL of the nanosheet solution resulted in immediate random aggregation of the nanosheets as a precipitate. The aggregated samples was washed with water and dried in air.

Table S1: Hydrolysis of starch over HNbMoO₆^a

Amount of starch / g	Amount of water / mL	Reaction time /h	Yields of products /mmol			Turnover number
			Glucose	Fructose	Hydroxymethyl furfural	
0.1	5	15	0.116	n.d.	n.d.	0.31
0.1	5	72	0.249	n.d.	0.136	1.01
0.1	2	15	0.170	0.053	0.238	1.21
0.2	2	15	0.324	0.033	0.191	1.44

^a Reaction conditions: starch (0.1 or 0.2g), H₂O (2 or 5 mL), catalyst (0.2g), 373 K.

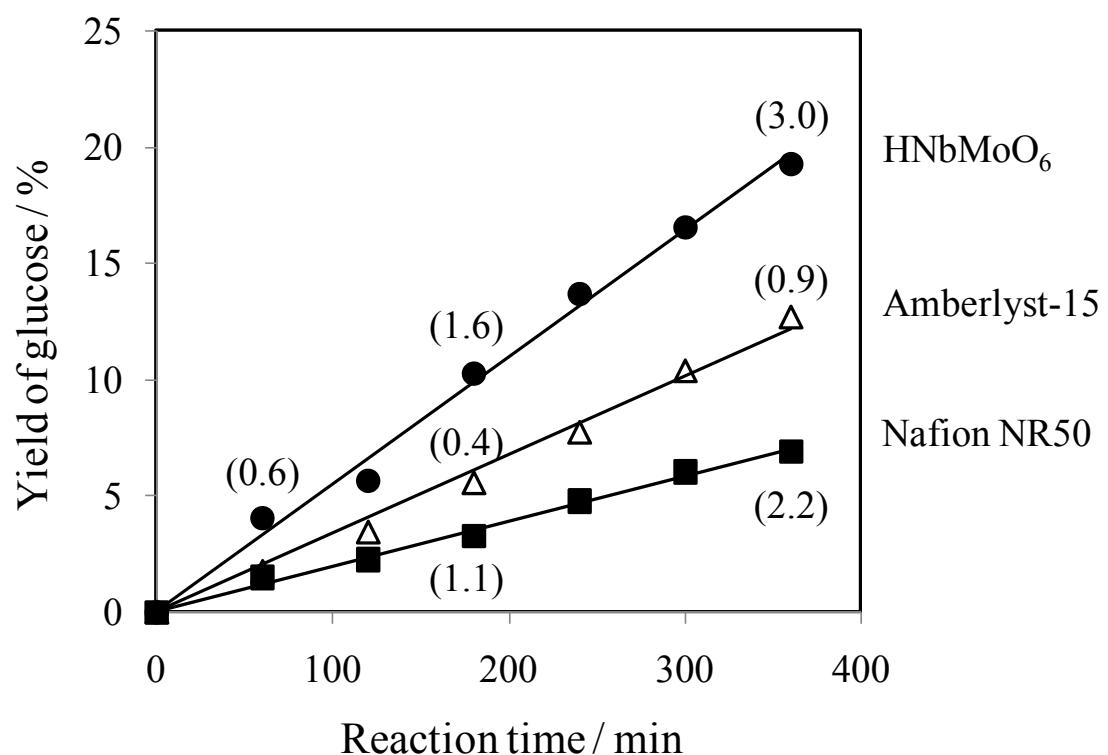


Figure S1: Time courses of cellobiose hydrolysis over layered HNbMoO₆, Amberlyst-15 and Nafion NR50. Values in parentheses are turnover number.

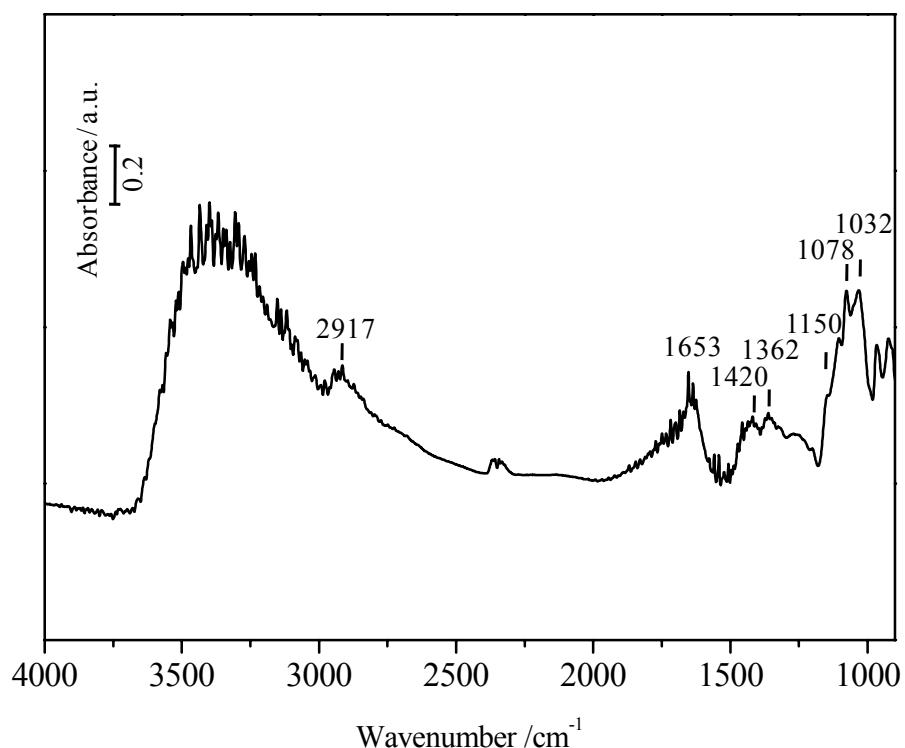


Figure S2: FT-IR spectrum for glucose-intercalated HNbMoO₆.
Assignments: 2917 cm⁻¹ (C-H stretch), 1420 cm⁻¹ (CH deformation), 1362 cm⁻¹ (CH deformation), 1150 cm⁻¹ (C-O stretch), 1078 cm⁻¹ (CO/CC stretch), 1032 cm⁻¹ (CO/CC stretch)