Supplementary Information

Fast ant facile dissolution of cellulose with tetrabutylphosphonium hydroxide containing 40 wt% water

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Experimental Details

Tetrabutylphosphonium hydroxide (TBPH) containing 60 wt% water was provided by Hokko Chemical Industry Co., Ltd., and we prepared several concentrated TBPH samples by evaporation. Tetrabutylammonium hydroxide (TBAH) containing 60 wt% water was purchased from Aldrich. Some concentrated TBAH samples were also prepared by evaporation. Microcrystalline cellulose (average molecular weight: 90000) was used as a standard cellulose for solubility tests. TBPH samples were put in a glass vial, and weighed cellulose powder, was added to the TBPH samples under stirring at 300 rpm at 25 °C. The dissolution time depended on the state of dispersion of the cellulose powder. The dissolution time used in this work is the shortest one because of the excellent dispersive property of the cellulose powder, based on several experiments for each case.

X-ray diffraction pattern was obtained using a Rigaku RINT-2500 diffractometer with Cu Ka radiation. The decomposition temperatures (T_{dec}) were determined by thermogravimetric analysis (TG/DTA 7200, Seiko Instruments) from 25 °C to 400 °C at a heating rate of 10 °C/min. The T_{dec} was assumed to be the temperature at the 10% weight loss on the TG chart. Infrared spectra were obtained by fourier transform infrared spectroscopy (FT/IR-4200, JASCO) with KBr methods. Molecular weight distribution of cellulose was determined with high performance ionic liquid chromatography (HPILC) with ionic liquid, 1-ethyl-3-methylimidazolium methylphosphonate, as an eluent. Cellulose powder or pullulan was added to the ionic liquid at 0.5 wt%, and chromatographs were obtained with flow rate of 0.01 ml min^{-1}. The structural change of TBPH containing water and dissolving mechanism were analysed with ^{1}H- and ^{13}C-NMR (α-400, JEOL) used double-chamber NMR tube with D_2O. The molecular weight was determined by fast atom bombardment mass spectrometry (FAB-MS) (MS700, JEOL).

Supplementary figures

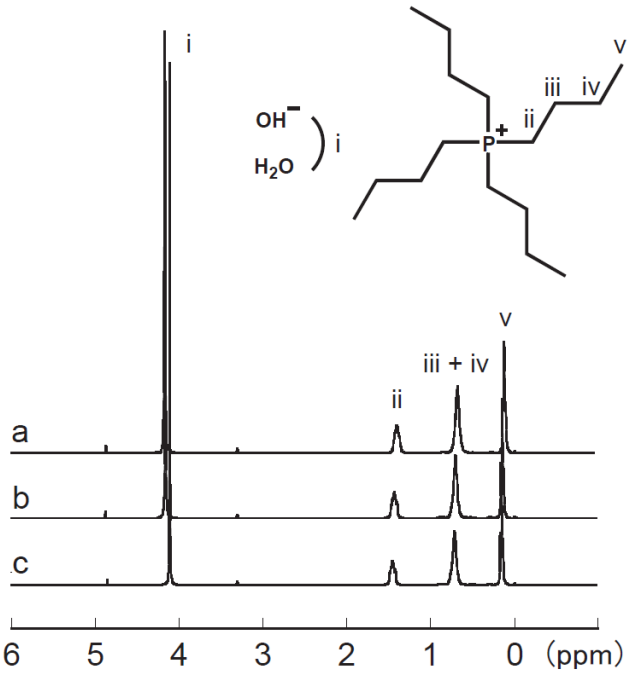

Fig. S1 ^{1}H-NMR spectra of TBPH containing 40 wt% water with 10 wt% cellulose (a), with 5 wt% cellulose (b), without cellulose (c).

Table S1 ^{1}H-NMR chemical shifts (δ (ppm) relative to TMS) of TBPH containing 40 wt% water with 10 wt% cellulose (a), with 5 wt% cellulose (b), without cellulose (c).

	δ (ppm)			
	i	ii	iii + iv	v
a	4.17	1.40	0.68	0.12
b	4.16	1.44	0.70	0.14
c	4.11	1.45	0.71	0.15

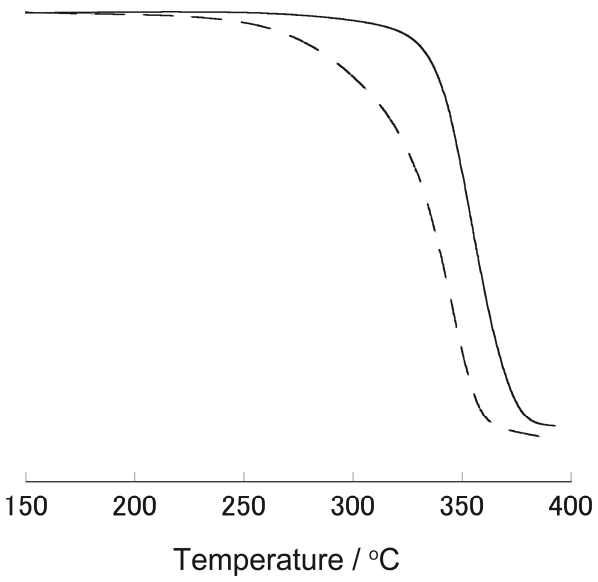

Fig. S2 TG/DTA measurements of cellulose before (solid line) and after (dashed line) dissolution with TBPH containing 40 wt% water.

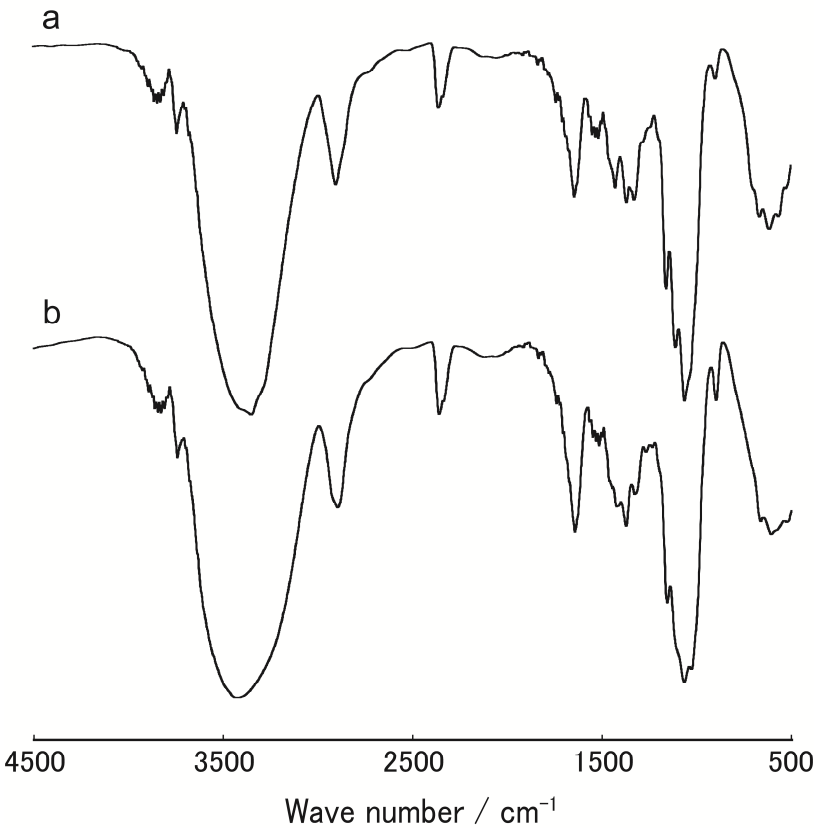

Fig. S3 FT-IR spectra of cellulose before (a) and after (b) dissolution with TBPH containing 40 wt% water.

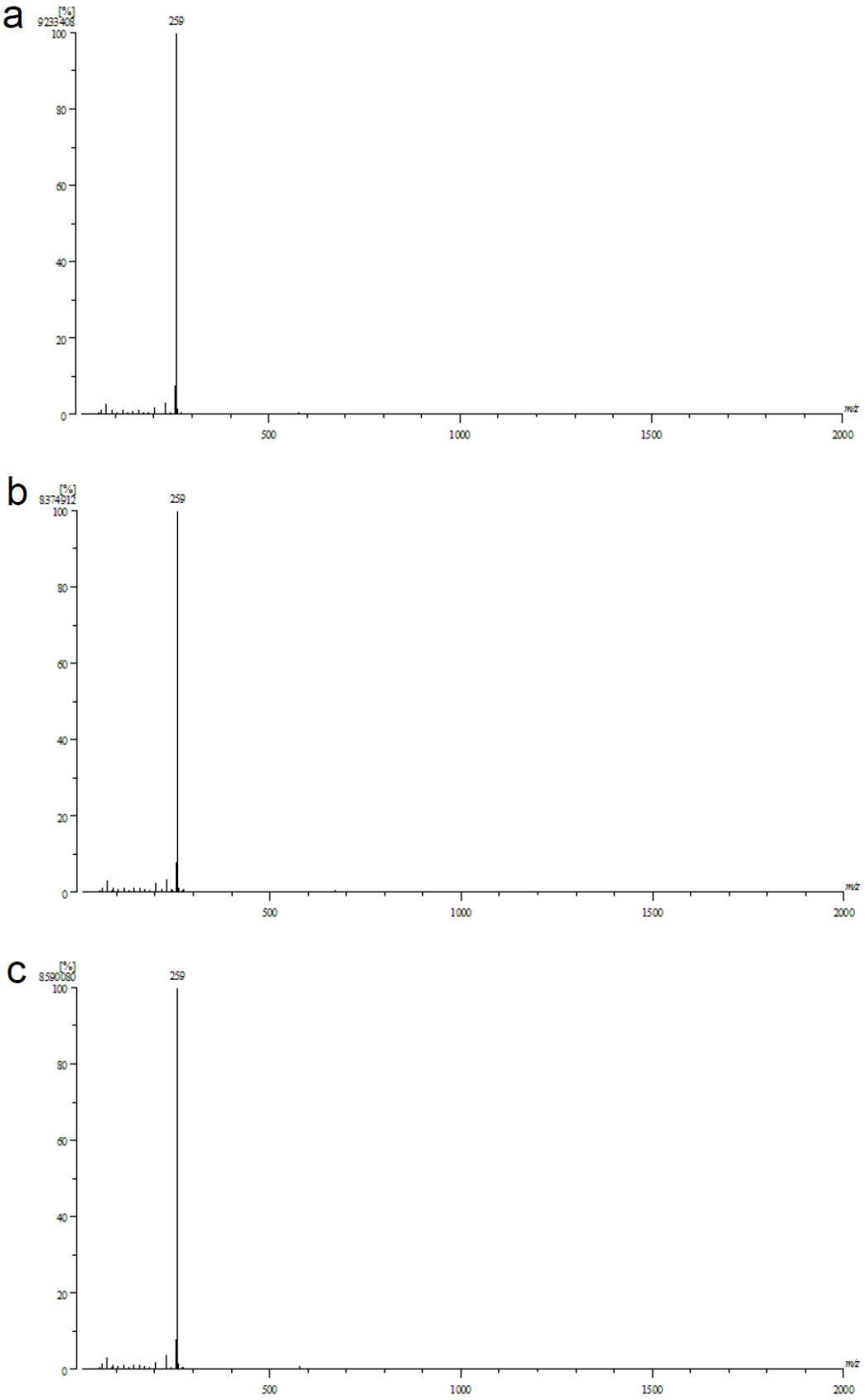

Fig. S4 FAB-MS spectra of TBPH containing 40 wt% water before treatment (a), after heating at 50 ^{o}C for 3h (b) and after cellulose dissolution process (c). (Molecular weight of tetrabutylphosphonium cation: 259.43)

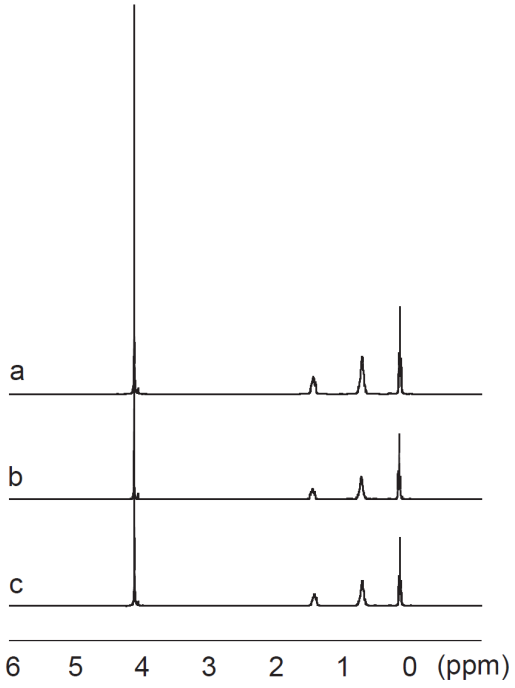

Fig. S5 ^{1}H-NMR spectra of TBPH containing 40 wt% water before treatment (a), after heating at 50 ^{o}C for 3h (b) and after cellulose dissolution process (c).

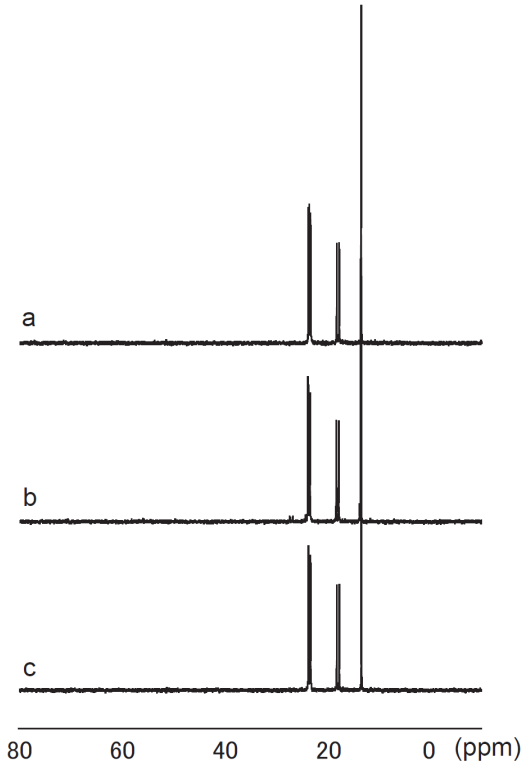

Fig. S6 ^{13}C-NMR spectra of TBPH containing 40 wt% water before treatment (a), after heating at 50 ^{o}C for 3h (b) and after cellulose dissolution process (c).