Supplementary material (ESI) for Journal of Materials Chemistry

Electronic Supplementary Information

Interlayer modification of a layered polysilicic acid H-octosilicate (H-RUB-18) with methanol : Formation of a highly ordered organosilicate nanohybrid

Shosuke Kiba,[a,c,d] Tetsuro Itagaki,[b] Teruyuki Nakato[c] and Kazuyuki Kuroda[a,b,d]*

[a]*Department of Nano-Science and Nano-Engineering, Faculty of Science & Engineering, Waseda University, 3-4-1 Ohkubo, Shinjuku-ku, Tokyo 169-8555, Japan.*
[b]*Department of Applied Chemistry, Faculty of Science & Engineering, Waseda University, 3-4-1 Ohkubo, Shinjuku-ku, Tokyo 169-8555, Japan.*
[c]*Institute of Symbioti Science and Technology, Tokyo University of Agriculture and Technology, 2-24-16 Nakacho, Koganei-shi, Tokyo 184-8588, Japan.*
[d]*Kagami Memorial Research Institute for Materials Science and Technology, Waseda University, 2-8-26 Nishiwaseda, Shinjuku-ku, Tokyo 169-0051, Japan.*
* kuroda@waseda.jp

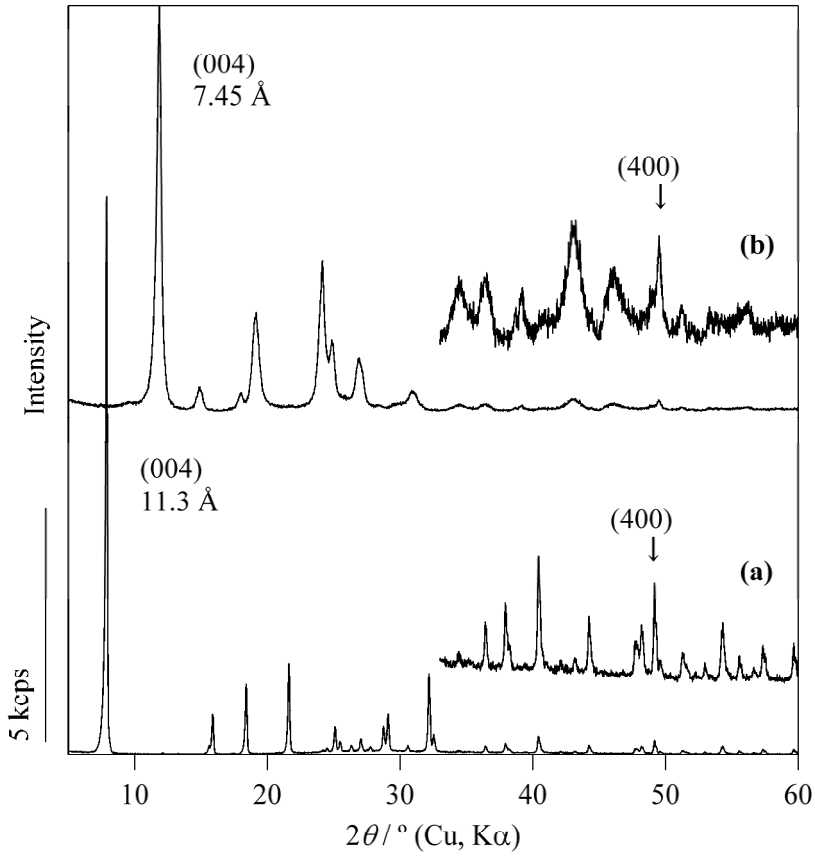

Fig. S1 XRD patterns of **(a)** Na-octosilicate and **(b)** H-octosilicate.

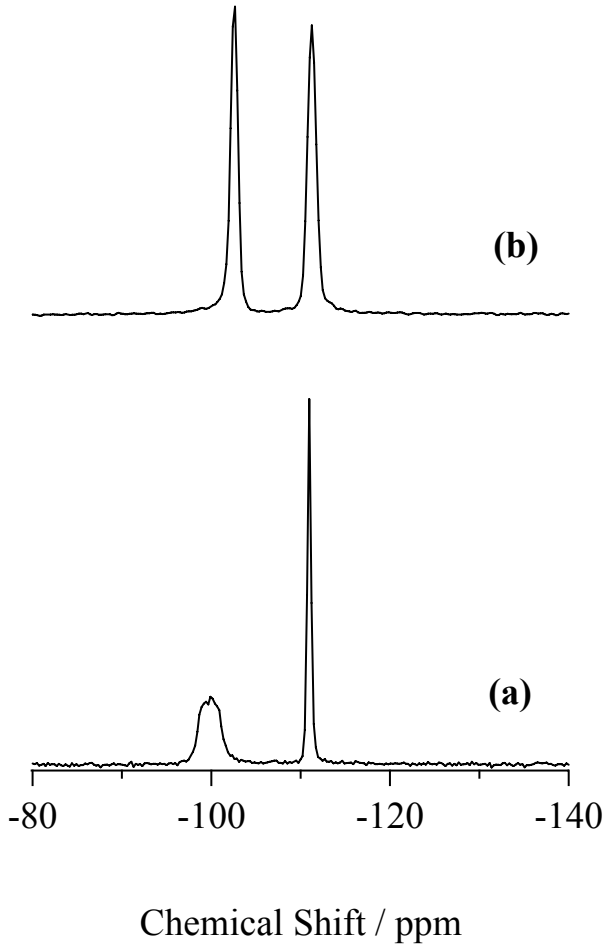

Fig. S2 ^{29}Si HD/MAS NMR spectra of **(a)** Na-octosilicate and **(b)** H-octosilicate.

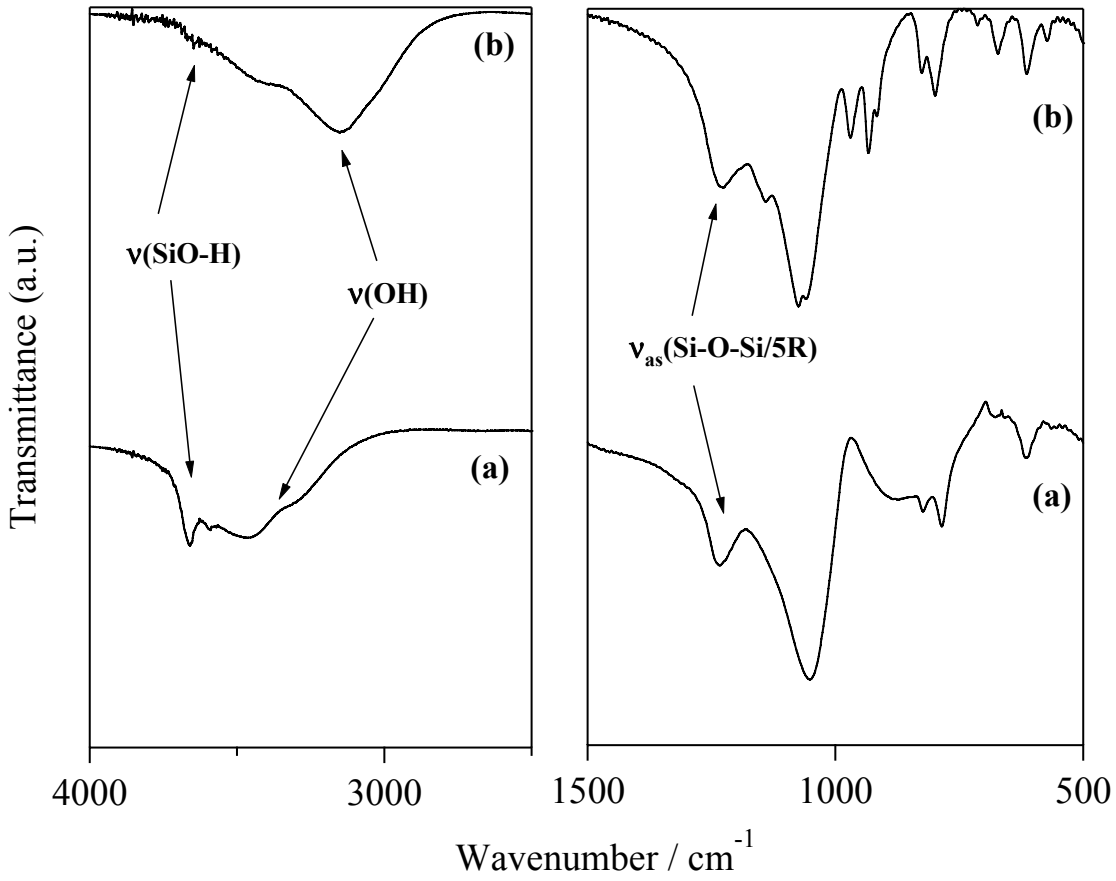

Fig. S3 IR spectra of **(a)** Na-octosilicate and **(b)** H-octosilicate.

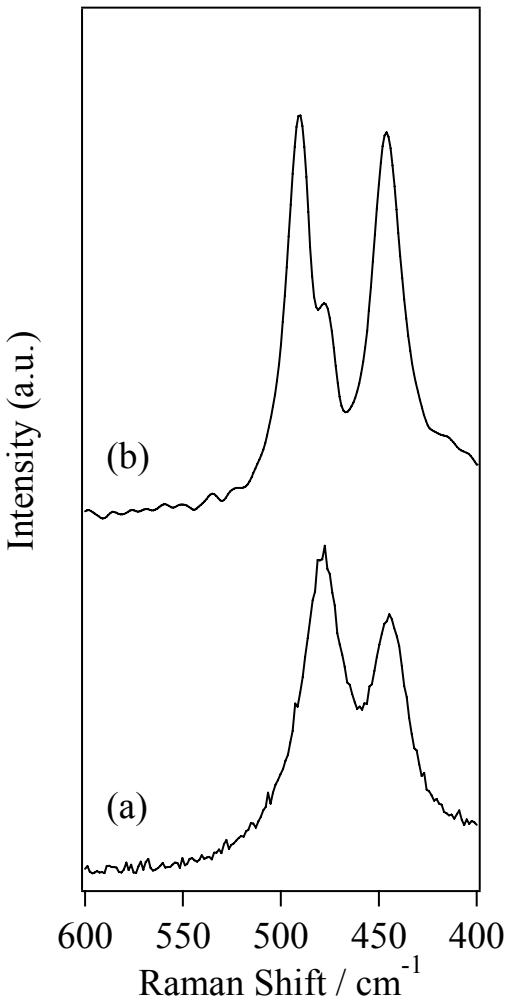

Fig. S4 Raman spectra of **(a)** Na-octosilicate and **(b)** H-octosilicate.

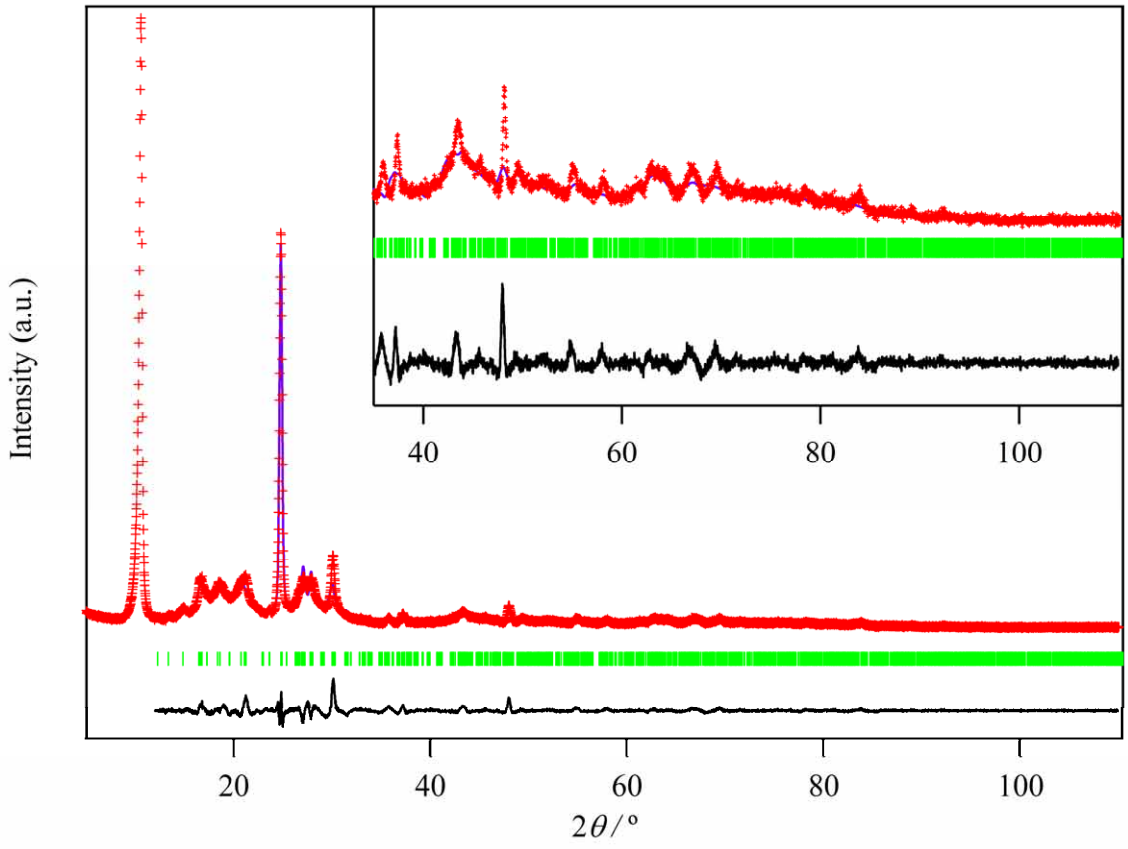

Fig. S5 Experimental **(red, "+" marks)** and calculated **(purple, solid line)** XRD patterns of methoxylated octosilicate, and the observed reflections **(green)** and difference **(black)** from the Rietveld refinement.

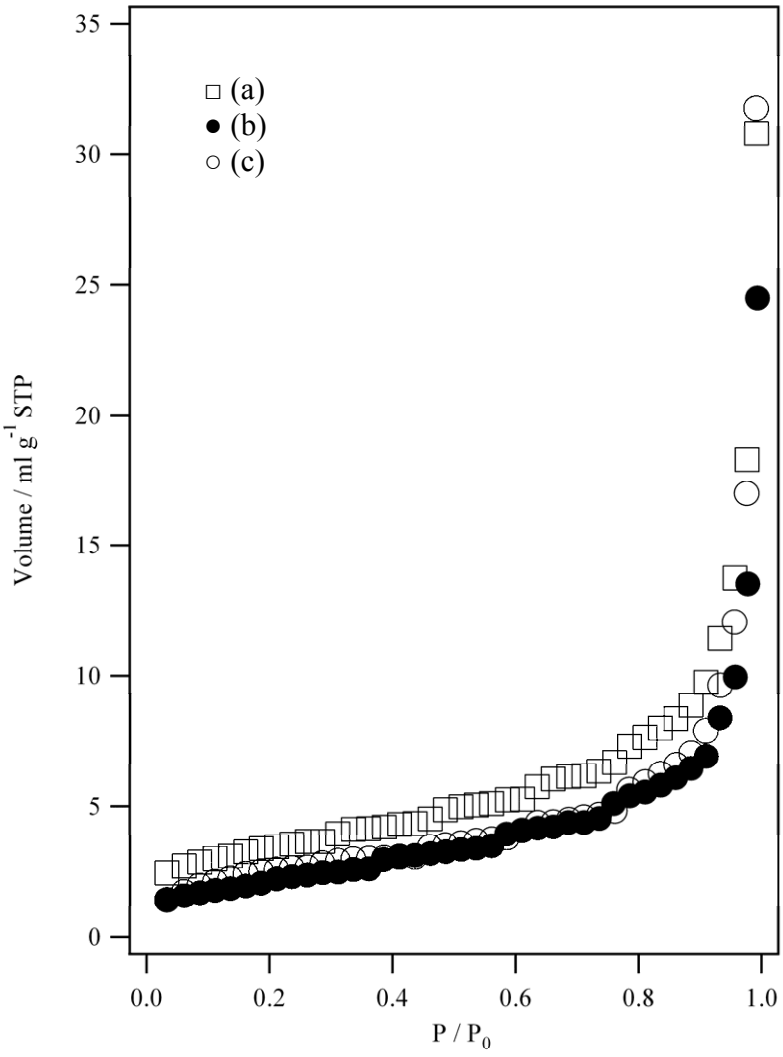

Fig. S6 N_2 adsorption isotherms of **(a)** H-octosilicate and methoxylated products with the grafting degrees of **(b)** 0.95 and **(c)** 0.42.

Table S1 Crystal data and refinement details for methoxylated octosilicate.

Formula	$Si_8O_{14}(OCH_3)_4$
M	572.8
Symmetry	Monoclinic
Space group	No.9, *Cc*
A /Å	10.654(8)
B /Å	10.72(8)
C /Å	21.447(5)
α /°	90
β / °	128.447
γ /°	90
Z	2
Pattern range, 2θ/°	12.00-110.00
Step scan increment, 2θ/°	0.005
Step scan time /s	2
Number of data	19600
Number of reflection	2446
Number of structural parameter	60
Number of profile parameter	10
Polynomial order of background coefficients	30
*R*p /%	7.59
*R*wp /%	10.5

Table S2 Refined structural parameters of methoxylated octosilicate.

Atom	Site	Occupancy	*x*	*Y*	*Z*	*U* /Å^2
C1	4a	1	-1.539(7)	0.304(8)	-0.696(4)	0.05
C2	4a	1	-0.344(9)	0.068(16)	0.282(7)	=*U*(C1)
C3	4a	1	-1.749(7)	-0.079(4)	-0.787(2)	=*U*(C1)
C4	4a	1	-3.015(4)	0.203(13)	-1.800(6)	=*U*(C1)
O1	4a	1	-2.652(5)	0.025(8)	-1.454(6)	0.02
O2	4a	1	-1.21(8)	0.063(4)	-0.412(7)	=*U*(O1)
O3	4a	1	-0.452(9)	0.164(4)	0.091(11)	=*U*(O1)
O4	4a	1	-1.845(6)	0.227(3)	-0.910(9)	=*U*(O1)
O5	4a	1	-0.607(9)	0.154(2)	-0.069(13)	=*U*(O1)
O6	4a	1	-2.13(6)	0.267(2)	-1.062(5)	=*U*(O1)
O7	4a	1	-2.891(5)	0.109(3)	-1.603(5)	=*U*(O1)
O8	4a	1	-1.421(7)	0.003(8)	-0.573(9)	=*U*(O1)
O9	4a	1	-1.555(4)	0.315(4)	-0.768(3)	=*U*(O1)
O10	4a	1	-1.17(8)	0.158(6)	-0.512(8)	=*U*(O1)
O11	4a	1	-0.492(9)	0.073(5)	0.197(2)	=*U*(O1)
O12	4a	1	-2.954(5)	-0.064(3)	-1.533(2)	=*U*(O1)
O13	4a	1	-1.646(7)	-0.139(7)	-0.707(6)	=*U*(O1)
O14	4a	1	-2.107(6)	0.080(6)	-0.972(9)	=*U*(O1)
O15	4a	1	-2.918(5)	0.255(4)	-1.718(8)	=*U*(O1)
O16	4a	1	-0.384(9)	0.330(5)	0.020(7)	=*U*(O1)
O17	4a	1	-1.742(7)	-0.044(9)	-1.162(16)	=*U*(O1)
O18	4a	1	-0.835(8)	-0.439(19)	-0.340(5)	=*U*(O1)
Si1	4a	1	-0.848(8)	-0.059(5)	-0.018(7)	0.01
Si2	4a	1	-2.229(6)	-0.034(7)	-0.993(6)	=*U*(Si1)
Si3	4a	1	-1.008(8)	-0.159(7)	-0.494(5)	=*U*(Si1)
Si4	4a	1	-2.526(5)	-0.247(5)	-1.490(9)	=*U*(Si1)
Si5	4a	1	-1.736(7)	0.351(9)	-0.858(2)	=*U*(Si1)
Si6	4a	1	-0.483(16)	0.04(7)	0.124(7)	=*U*(Si1)
Si7	4a	1	-1.604(9)	-0.062(7)	-0.628(3)	=*U*(Si1)
Si8	4a	1	-2.801(6)	0.161(5)	-1.637(6)	=*U*(Si1)

Table S3 Bond lengths in the methoxylated octosilicate.

Atoms	Length (Å)	Atoms	Length (Å)
O1- Si1	1.680	O9- C1	1.455
O1- Si6	1.671	O10- Si2	1.626
O2- Si2	1.650	O10- Si4	1.649
O2- Si5	1.633	O11- C2	1.496
O3- Si4	1.652	O12- Si1	1.631
O3- Si6	1.628	O12- Si3	1.635
O4- Si3	1.704	O13- C3	1.490
O4- Si5	1.658	O14- Si3	1.633
O5- Si4	1.670	O15- C4	1.487
O5- Si7	1.627	O16- Si1	1.618
O6- Si3	1.673	O16- Si4	1.647
O6- Si8	1.685	O17- Si7	1.635
O7- Si1	1.669	O17- Si8	1.628
O7- Si8	1.631	O18- Si5	1.632
O8- Si2	1.696	O18- Si6	1.649
O8- Si7	1.671		