Supplementary Material (ESI) for Journal of Materials Chemistry

New insights into the encapsulation and stabilization of heteropolyacids inside the pore walls of mesostructured silica materials

Véronique Dufaud,*[1] Frédéric Lefebvre,*[2] Gerald P. Niccolai[3]and Mimoun Aouine[4]

Supporting Information for the Journal of Material Chemistry

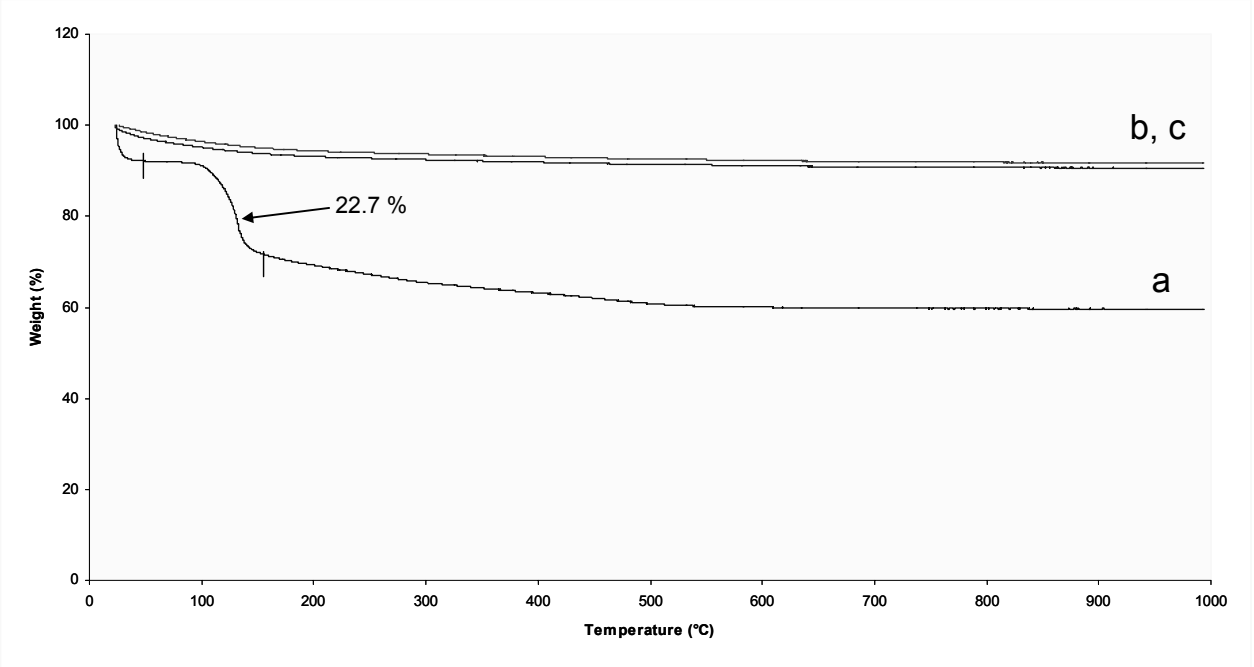

Fig. S1 Thermogravimetric analysis of (a) **2**; (b) and (c) **2-C** and **2-CE**

[1] Laboratoire de Chimie, UMR 5182 CNRS - Ecole Normale Supérieure de Lyon, 46 allée d'Italie, 69364 Lyon cedex 07, France ; vdufaud@ens-lyon.fr
[2] Chimie, Catalyse, Polymères et Procédés UMR 5265 CNRS-CPE Lyon, 43 bd du 11 Novembre 1918, 69622 Villeurbanne cedex, France; lefebvre@cpe.fr
[3] ICAR, UMR 5191 ENS LSH, 15 parvis Descartes, 69342 Lyon cedex 07, France.
[4] Institut de Recherches sur la Catalyse et l'Environnement de Lyon, 2 avenue Albert Einstein, 69626 Villeurbanne cedex, France.

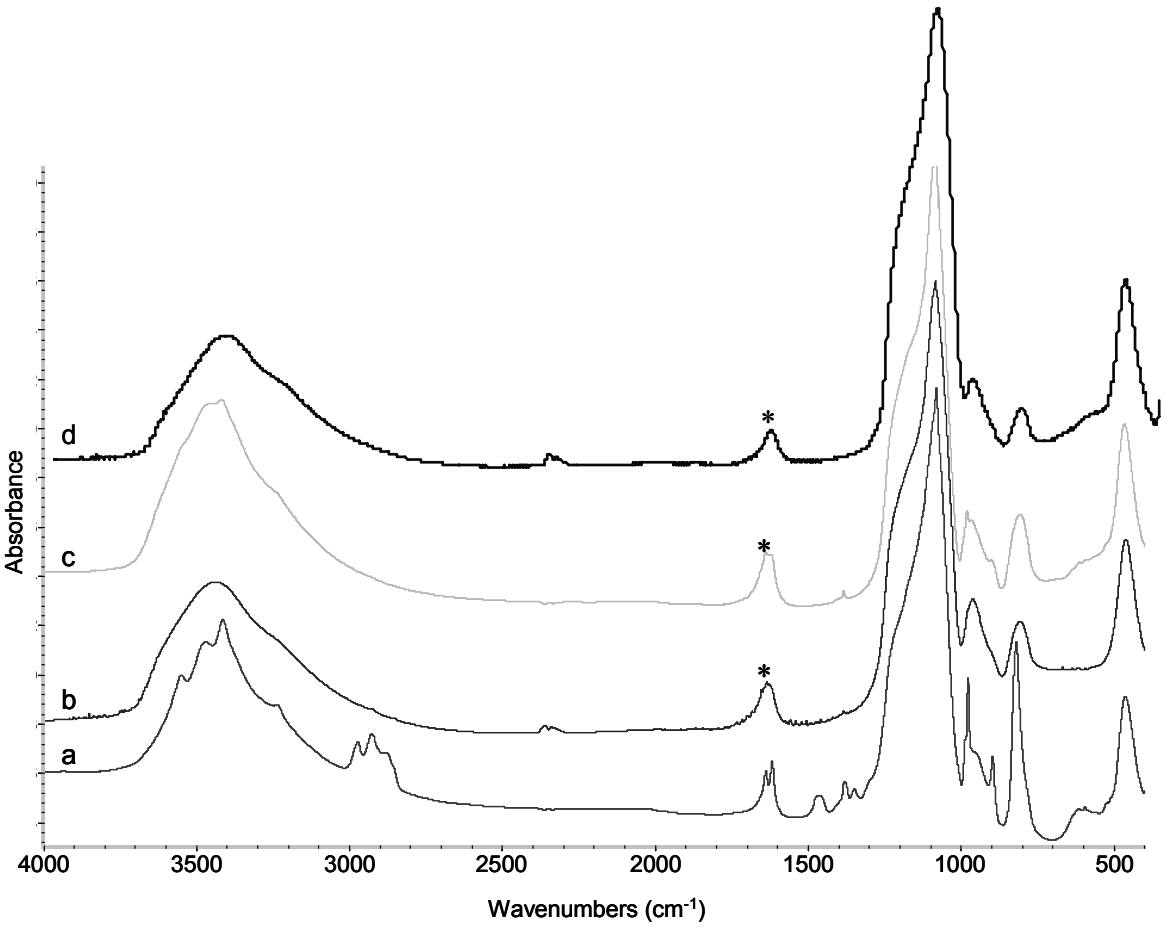

Fig. S2 FT-IR spectra of (a) **2;** (b) **2-C;** (c) **2-CE**; (d) **2-E**

* at ca. 1630 cm^{-1} denotes for adsorbed residual water

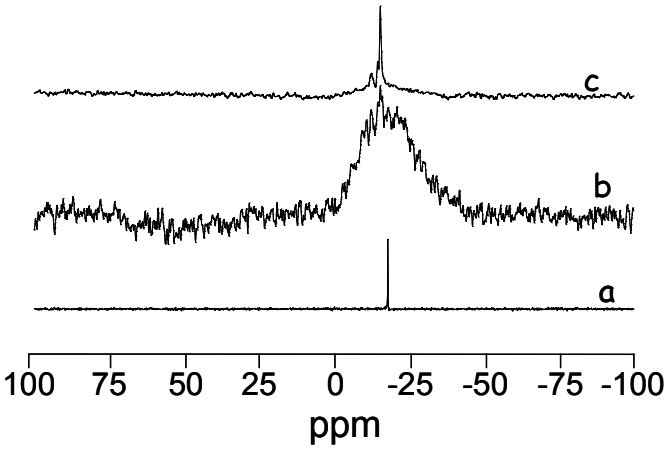

Fig. S3 MAS ^{31}P NMR spectra of (a) **2;** (b) **2-C;** (c) **2-CE** (second lot, W 28.5%$_{wt}$)

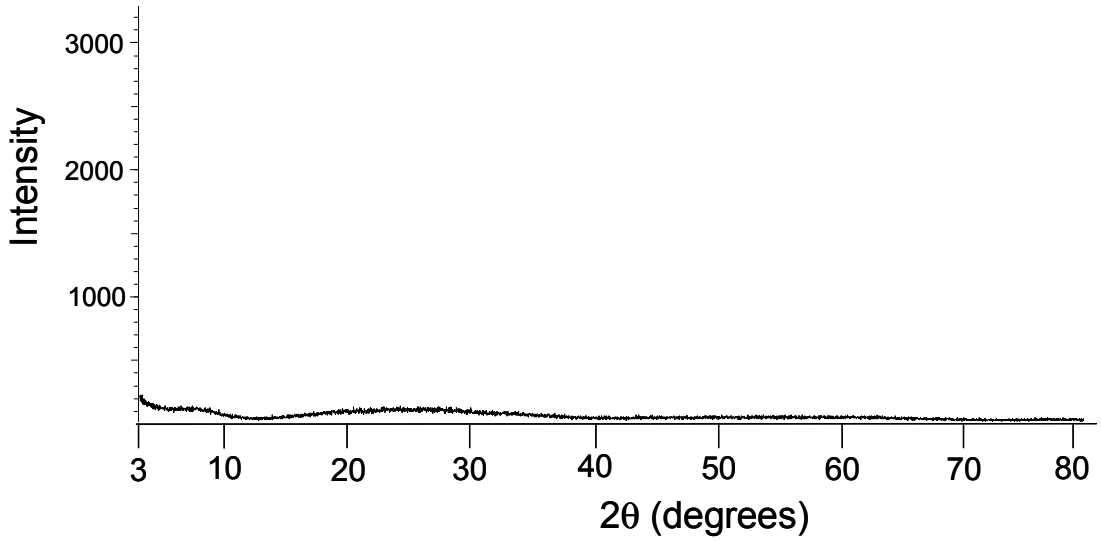

Fig. S4 XRD pattern of **2-CE** in the 3° to 80° (2θ scale) region

Typical TEM/EDX analysis on $H_3PW_{12}O_{40}$ encapsulated into SBA-15 silica material (28.55 $\%_{wt}$ W ≅ 9.3 mol W per 100 atom Si) (2-CE second lot)

Sampling #	Si (atom)	W (atom)
1	100	12.80
2	100	14.93
3	100	14.74
4	100	11.6

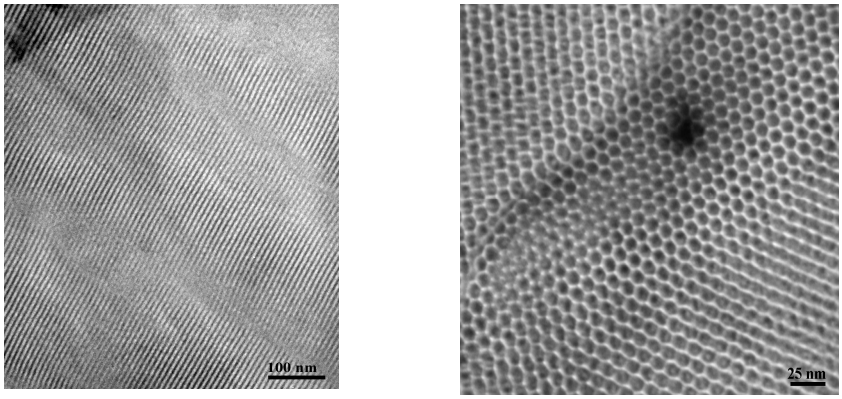

Fig. S5 TEM micrograph of **2-CE** (second lot), in the direction perpendicular to the pore axis (left) and along the direction of the pore axis (right)

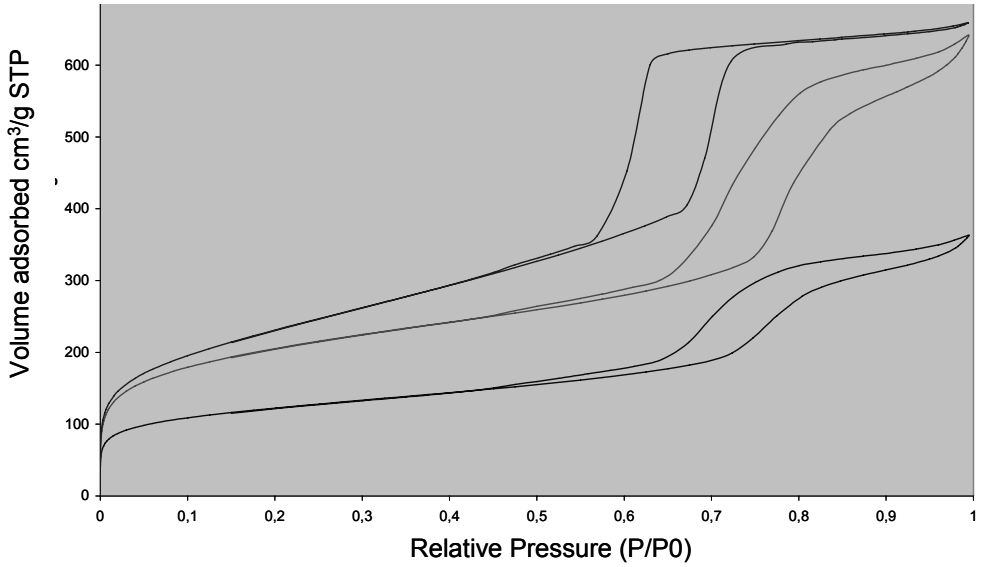

Fig. S6 Nitrogen adsorption/desorption isotherm of **2-CE** (bottom) ; **2-E** (middle) ; **1-CE** (top)

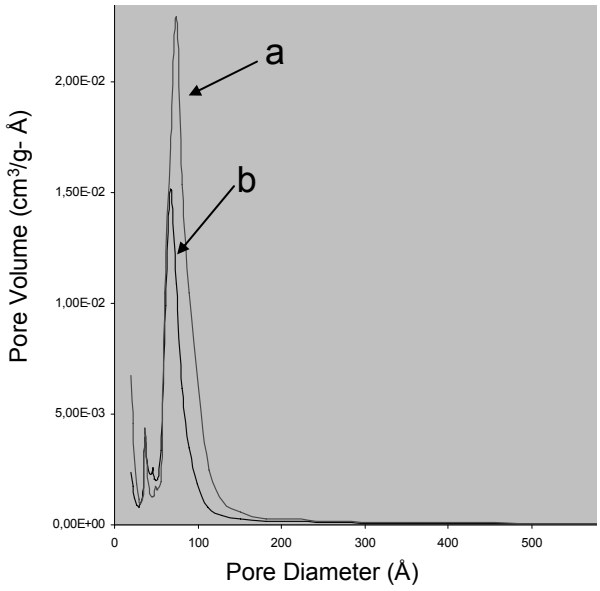

Fig. S7 Pore size distribution (from the BJH calculations) of (a) **2-E** ; (b) **2-CE**

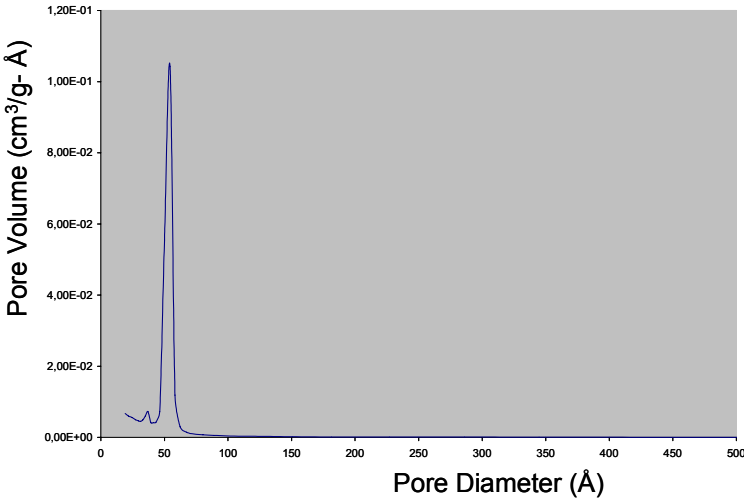

Fig. S8 Pore size distribution (from the BJH calculations) of **1-E**

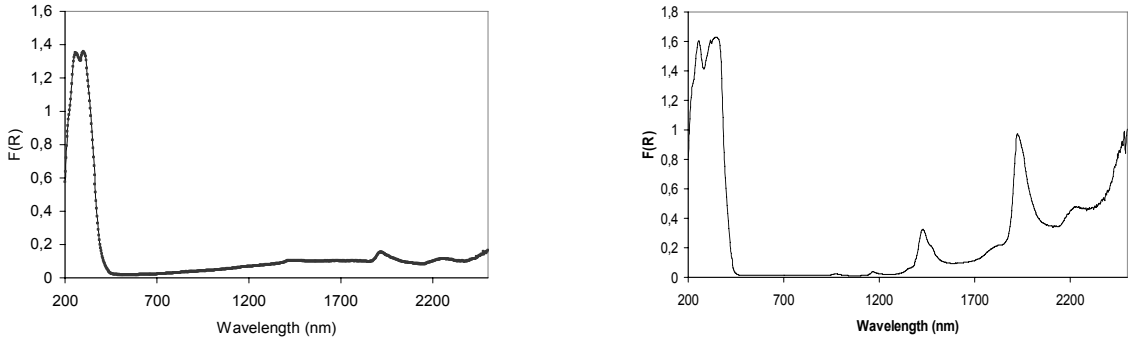

Fig. S9 DR UV-Vis of **2-CE** (left) and of $H_3PW_{12}O_{40}$ (right)

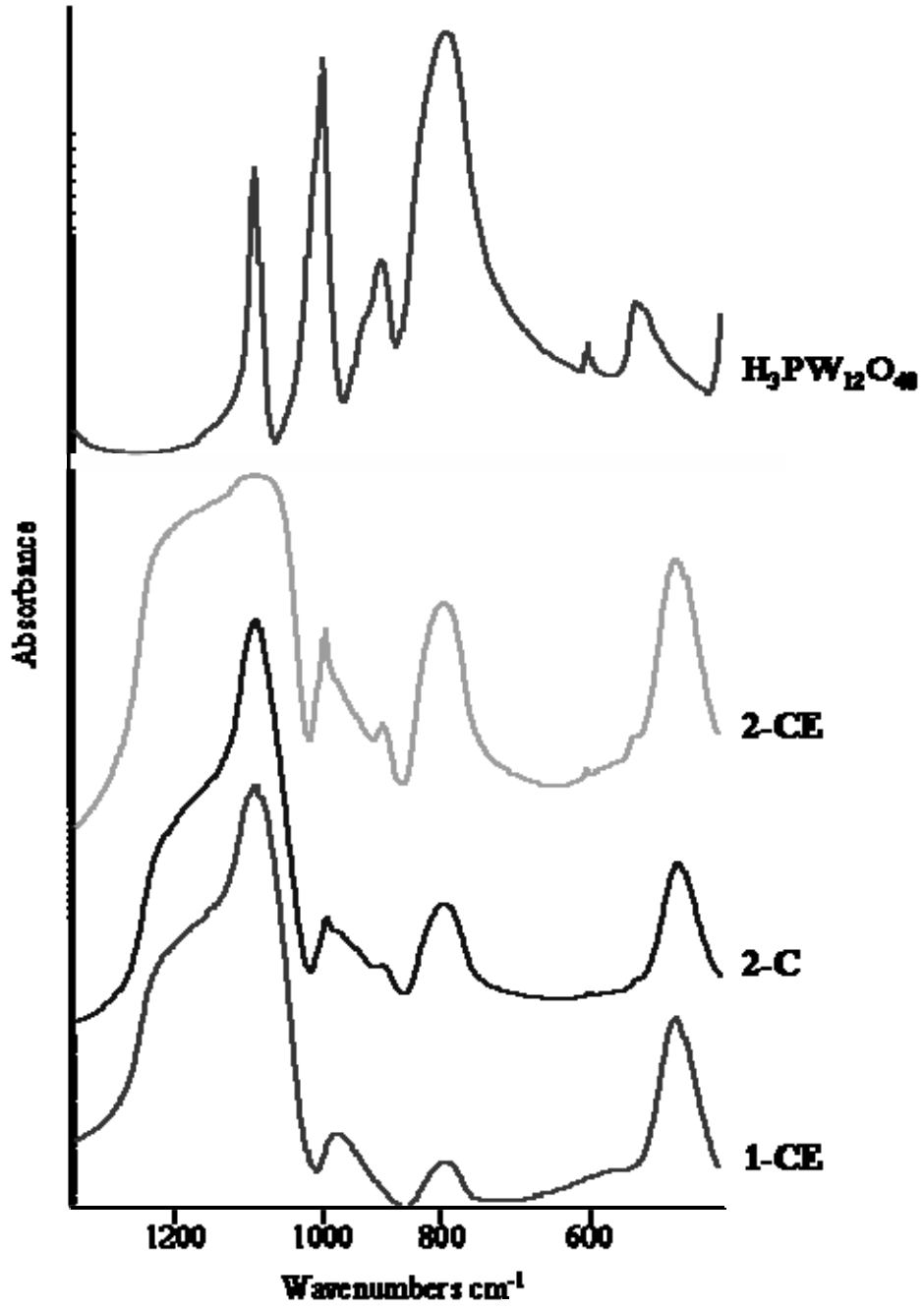

Fig. S 10 Infrared spectra of $H_3PW_{12}O_{40}$, **2-CE**, **2-C**, and **1-CE**. One should note that for **2-C** and especially **2-CE**, one can discern the characteristic absorbances of the molecular species (top) around 630 and 980 cm^{-1} (other characteristic peaks are masked by strong silica absorbances). Such peaks are lacking in the spectrum of **1-CE**.

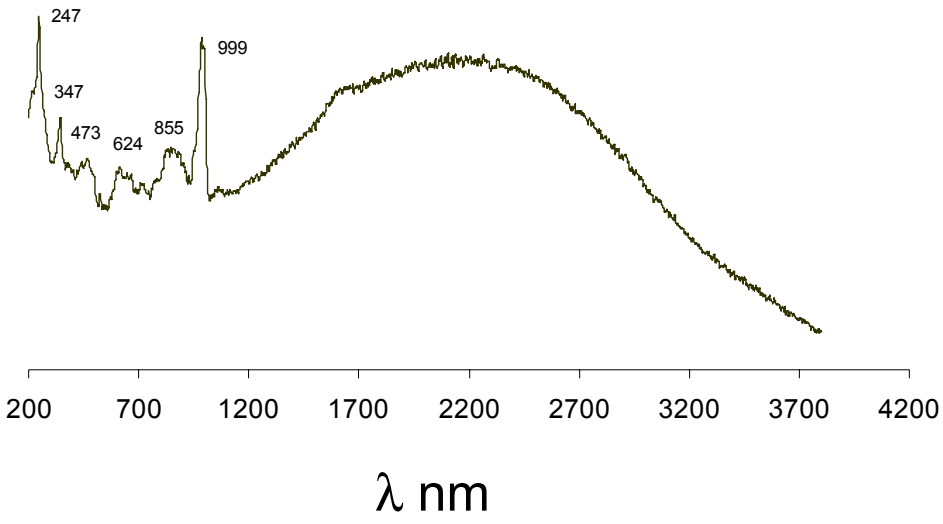

Fig. S 11 Raman spectroscopy of $H_3PMo_{12}O_{40}$ encapsulated in SBA-15 type silica after calcination and extraction