Supplementary ESI

Figure S1 shows the $^{29}$Si NMR spectra with signals representative of various silicon environments of the Mn and Qn silane moieties; however, Mn resonance peaks are representative of silicon matrix directly bonded to organic species. Two main resonance peaks at -110.1 ppm and at -101 ppm, correspond to Si(OSi)$_4$ (Q4) and (HO)Si(OSi)$_3$ (Q3) silicate species, respectively, were observed. In fact, with incorporation of TMS groups into the silica NTs, the Q3 peak decreased substantially, and a new peak (M1) appeared at 13 ppm due to silicon bonded to the TMS, as we recently reported [32]. Moreover, the increase in the intensity of the siloxane groups’ peak (Q4) (data not shown) indicates that Si–OH sites (corresponding to silanediol and silanol groups) in the inner pores of the silica NTs underwent condensation with TMS, thus forming covalent linkages to the silica framework. The sharp signal of (M1) corresponds to the main component of the hydrophobic TMS silane, indicating that TMS molecules were located in close proximity of each other in a well-defined monolayer rather than a polymerized random multilayer.

![Fig. S1 $^{29}$Si nuclear magnetic resonance (NMR) spectra and a deconvolution of each peak of 3D mesocage TMS-silica NTs nanofilters.](image-url)
Supplementary ESII

The TG profile (Fig. S2) shows the gradual decrease in the weight of the TMS-silica NTs nanofilter up to ~20 wt% from 25-700 °C. The TG curve indicates three distinct stages of weight loss accompanied by a DTA exothermic peak in each of the three stages. First, the weight loss of 2.25 wt% before 150 °C might indicate the evaporation of physically adsorbed H₂O and remained ethanol during the dense silanization of TMS into silica NTs. Second, the weight decrease between 200 and 400 °C is related to the decomposition of organic TMS groups, which corresponds to one strong exothermic DTA peak around 200–400 °C. Thus, the weight loss of 13.25 wt% between 200 and 400 °C might be attributed to the decomposition of organic TMS moieties. Third, the weight loss of 2.9 wt% between 400 and 600 °C might be assigned the further condensation of the silica species (dehydroxylation).

Fig. S2 TG and DTA analyses of the 3D mesocage TMS-silica NTs nanofilter after complete etching of the alumina membranes by 5% H₃PO₄ solution at 25 °C for 10 h.