Template confined synthesis of Cu- or Cu$_2$O- doped SiO$_2$ aerogels from Cu(II)-containing composites by in-situ alcohothermal reduction

Weiwei Xu, Ai Du, Jun Tang, Peng Yan, Xiaoguang Li, Zhihua Zhang, Jun Shen, Bin Zhou*  
Shanghai Key Laboratory of Special Microstructure Materials and Technology, School of Physics Science and Engineering, Tongji University, Shanghai 200092, P. R. China.

Fig. S1. Adsorption-desorption isotherms of composite aerogels: (a) initial aerogels, and different reducing products: (b) ethanol, (c) ethylene glycol, (d) glycerol.

As shown in Fig. S1, the adsorption-desorption isotherms of initial Cu(II)-contained silica templates and correspond reducing products with different reducing agents (ethanol, ethylene glycol and glycerol) display similar multilayer adsorption behaviors. The total pore volumes of resulting samples were 2.775 cm$^3$·g$^{-1}$, 2.2 cm$^3$·g$^{-1}$, 4.16 cm$^3$·g$^{-1}$, 2.57 cm$^3$·g$^{-1}$, separately.
Fig. S2. EDS spectrum of Cu(II)-contained silica composite gels.

Fig. S3. EDS spectrum of resulting sample reduced by ethanol.

Fig. S4. EDS spectrum of resulting sample reduced by ethylene glycol.
In our work, the doping fraction of initial templates in the range from 1% to 20% ($n_{\text{Cu}}:n_{\text{Si}}$) could be accurately controlled via a co-gelation method. As shown in Fig. S5, EDS spectrum of Cu(II)-containing composite aerogels presented that the molar ratio of Cu to Si was about 19.95% (theoretical value 20%). During the subsequent alcohothermal process, small amount of generated Cu or Cu$_2$O nanoparticles released from the gel frameworks and dispersed in reducing agent. Therefore, the doping fractions of the resulting aerogels were partly decreased comparing with initial templates, and the loss ratio was increased with the reducibility enhanced of reducing agent. Consequently, the resulting ratio may be influenced by two factor: (1) the initial doping fraction of Cu(II)-containing templates, which could be accurately controlled using co-gelation method; (2) the reducing agent and reaction temperature, which could be adjusted according to need of reducing experiment.