

Supplementary Information

Synthesis:

The PS-PI diblock copolymer was synthesized using standard sequential anionic polymerization techniques. Styrene and isoprene were purified by treating with dibutyl magnesium for 4 h and *n*-butyl lithium for 4 h. Styrene was initiated by *sec*-butyllithium under an argon atmosphere and polymerized for 4 hours at 40 °C in cyclohexane, which was followed by isoprene addition, also polymerized for 4 hours at 40 °C. The polymerization was terminated with degassed methanol.

Crosslinking procedure:

The B μ E sample was placed in a Petri dish and contained in a closed glass jar. Sulfur monochloride (S₂Cl₂) was then added into the glass jar but outside the Petri dish. The S₂Cl₂ molecules were slowly absorbed into the B μ E sample by vapor phase transfer.

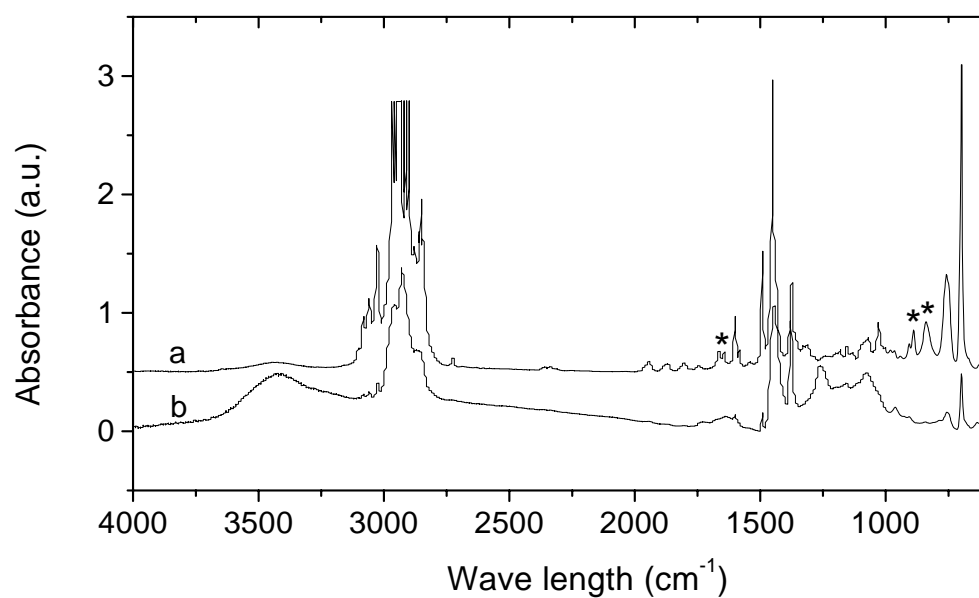
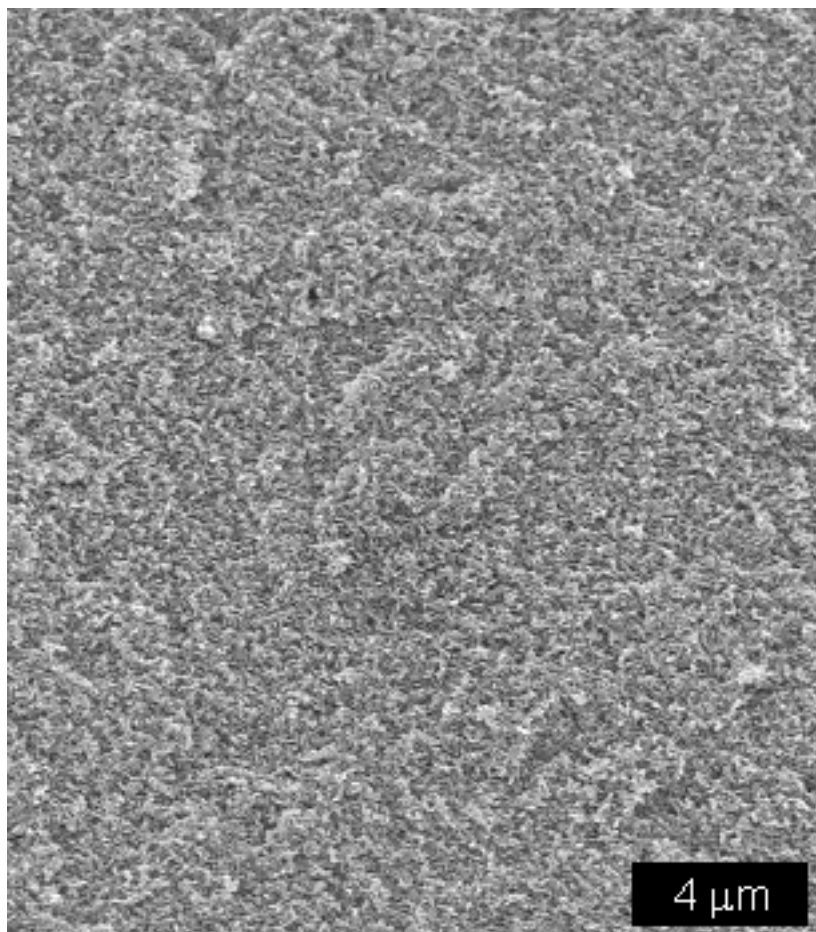
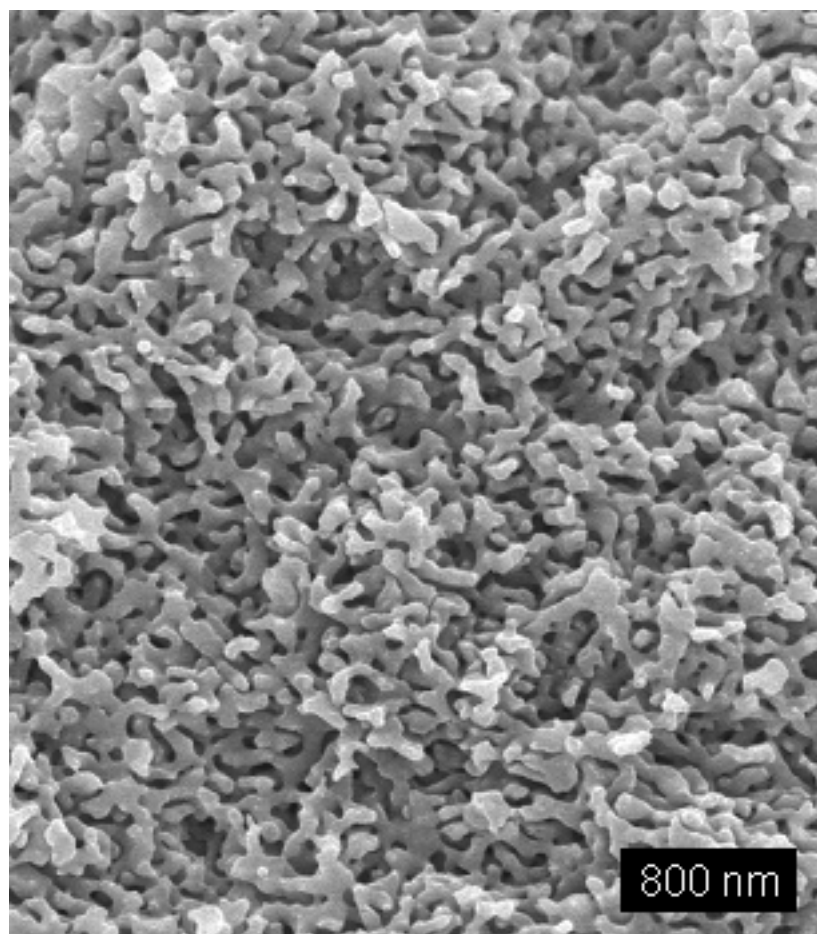


Figure S1. IR spectra of the BμE precursor (a) and the resultant nanoporous material (b). The * denotes the characteristic peaks from the double bonds of polyisoprene. The plots are shifted for clarity.

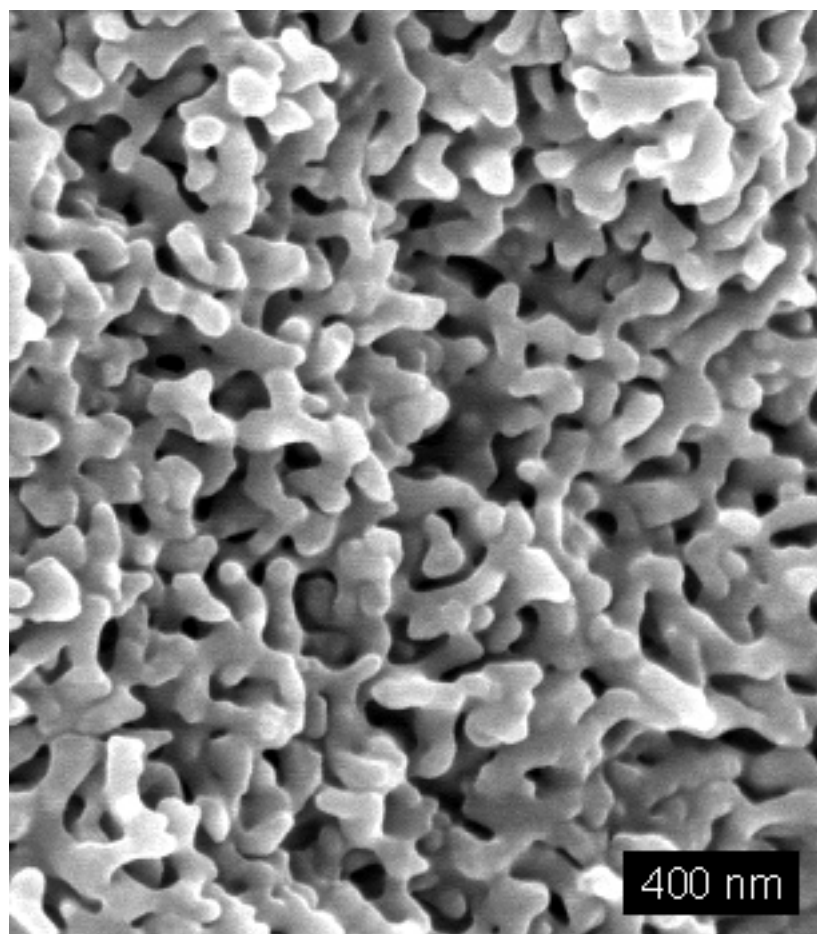


(a)

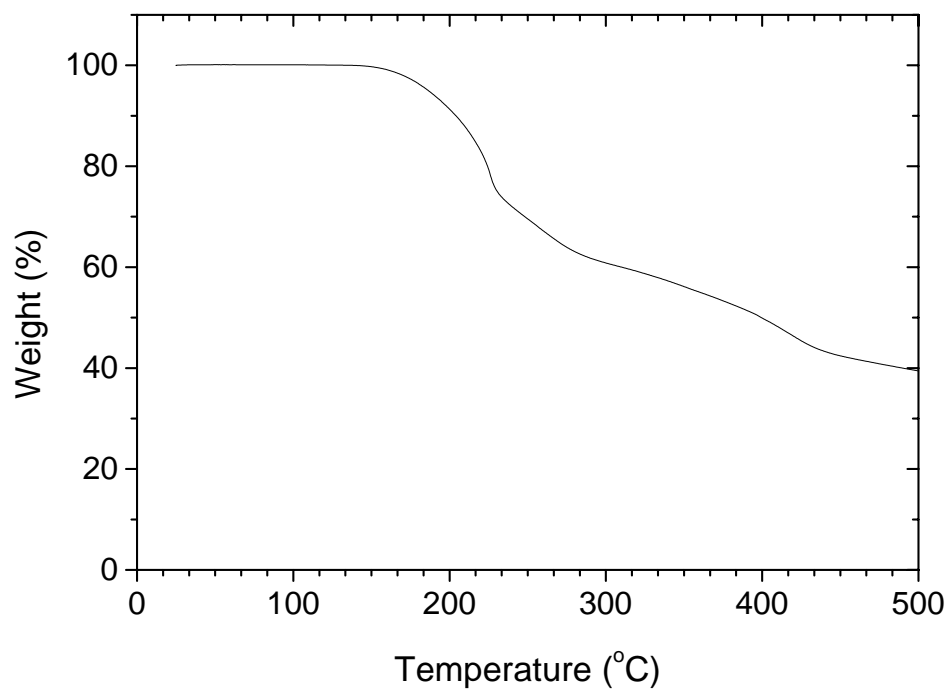


(b)

Figure S2. SEM images of the freeze-fractured surface of nanoporous material from the B μ E sample under two different magnifications: a) $\times 5,000$; b) $\times 25,000$.



(a)



(b)

Figure S3. (a) SEM image of the nanoporous material after being annealed at 200 °C for 3 hours under vacuum. (b) Thermogravimetric analysis of the nanoporous material.