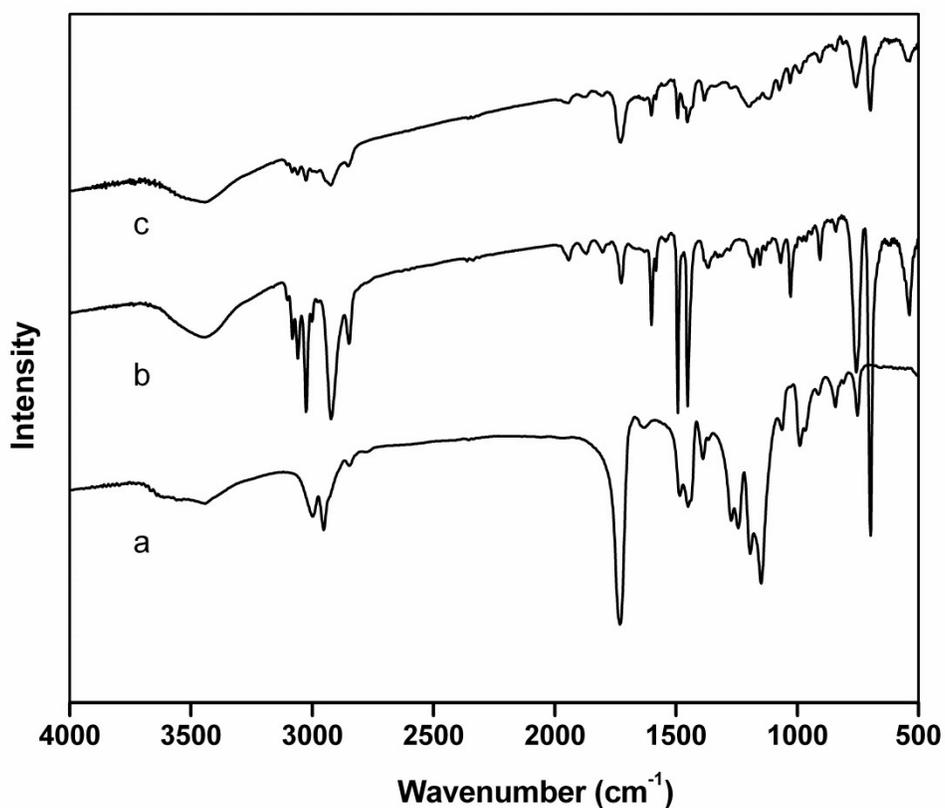


***Ex situ* Cu(0) nanoparticles mediated SET-LRP of methyl methacrylate/styrene-  
methyl methacrylate in biphasic toluene-water system**

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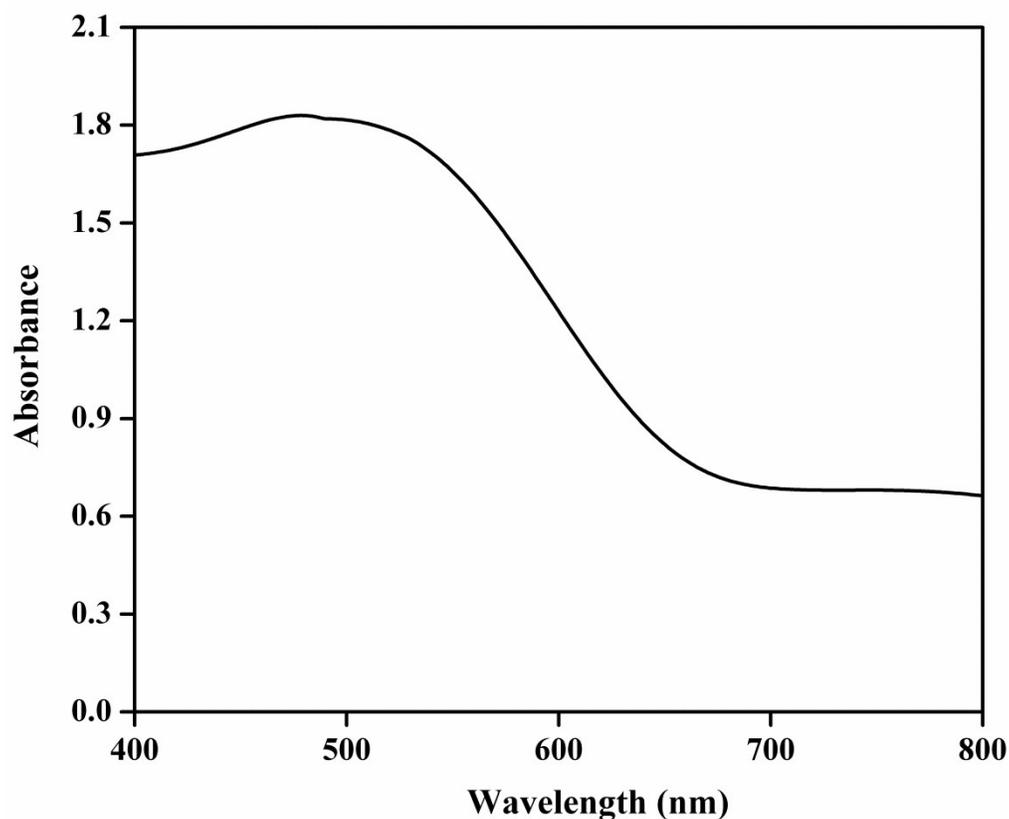
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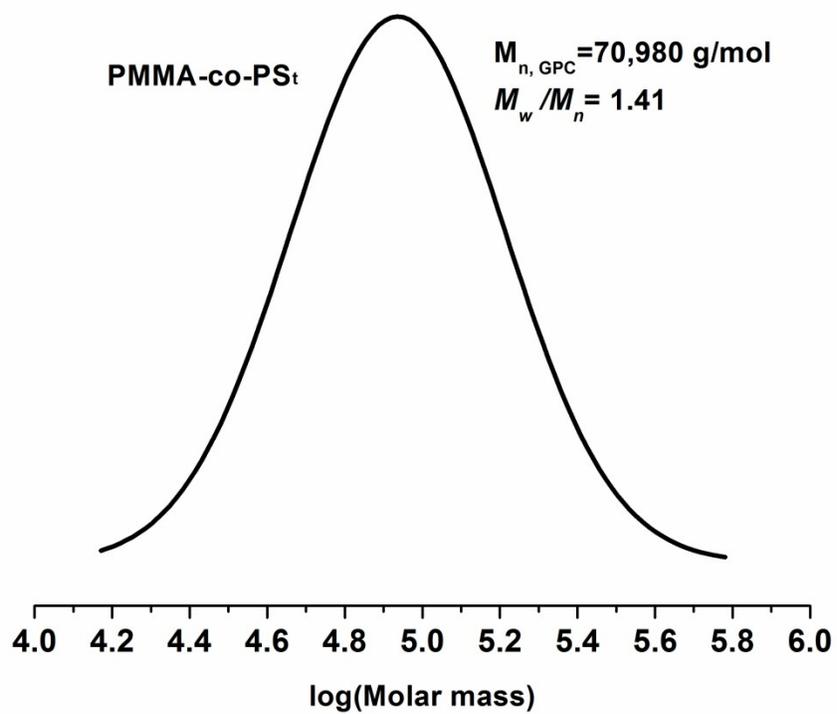
**Figure S1.** FTIR spectra of (a) PMMA synthesized by Cu(0) nps mediated SET-LRP. Reaction conditions:  $[MMA]_0/[EBriB]_0/[PMDETA]_0 = 100:4:5$ , Cu(0) nps = 28.5 ppm, MMA/toluene = 1:1 (v/v),  $[MMA]_0 = 4.70$  M, Temp = 70 °C, reaction time = 45 min, monomer conversion = 11%; (b) PS for comparison; (c) PMMA-*co*-PS prepared by SET-LRP using Cu(0) nps as catalyst and PMDETA as

ligand, initiated by EBriB. Reaction conditions:  $[St]_0/[MMA]_0/[EBriB]_0/[PMDETA]_0 = 100:100:3:2$ ,  $Cu(0)$  nps = 14.3 ppm, Temp = 70 °C, reaction time = 3 h, monomer conversion ~ 11%

FTIR spectrum of PMMA-*co*-PSt shows characteristics peaks of both MMA and St indicating the incorporation of MMA and St. Peaks at 3058 and 3025  $cm^{-1}$  are due to aromatic =C-H of styrene, 2921 and 2849  $cm^{-1}$  (aliphatic -C-H) of MMA and Styrene, 1491, 1448, 1600  $cm^{-1}$  (Phenyl stretching) and peak at 1731  $cm^{-1}$  is due to C=O of MMA.



**Figure S2.** UV-Vis spectra of supernatant solution containing Copper(0) nanoparticles, which was obtained after centrifugation of freshly synthesized copper nanoparticles at 8000 rpm for 15 min.



**Figure S3.** GPC curve of PMMA-co-PSt copolymer having narrow dispersity.