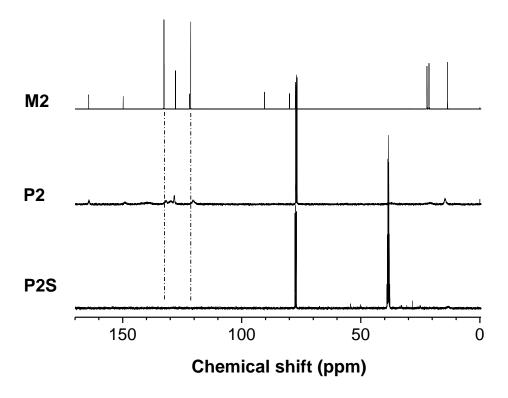
## Electronic Supplementary Information (ESI) for

## A new strategy of post-polymerization modification to prepare functionalized poly(disubstituted acetylenes)

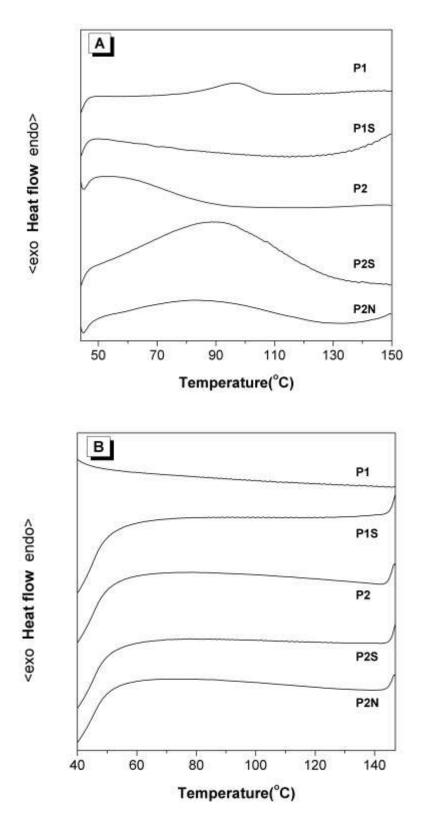
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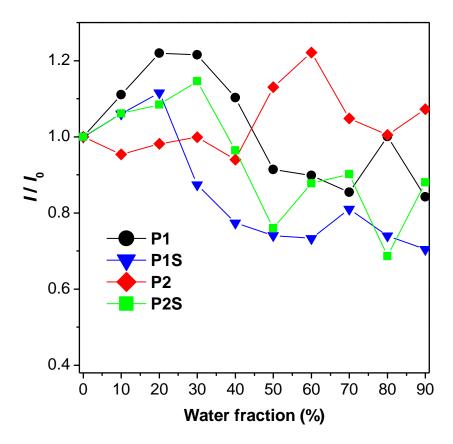
**Fig. S1** <sup>13</sup>C NMR spectra of **M2**, **P2** and **P2S**. The disappearance of the chemical shifts implies the elimination of the vinyl groups.

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**Fig. S2** Differential scanning calorimetric (DSC) curves for all polymer samples (**P1**, **P2**, **P1S**, **P2S**, and **P2N**), (A) and (B) are heating and cooling processes recpectively. Scanning rate: 20 °C/min; hold for 2.0 min at 150.00 °C.



**Fig. S3** Variation of the relative fluorescence intensity ( $I/I_0$ ) of the obtained poly(disubstituted acetylenes) (**P1**, **P2**, **P1S** and **P2S**) in THF/water mixtures with changing water fraction (by volume) from 0 to 90%, where I and  $I_0$  are the fluorescence intensity recorded for the samples in the pure THF solution. Polymer concentration: 10  $\mu$ M; Excitation wavelength: 280 nm.