

## Electronic Supplementary Material (ESI)

Pb<sup>2+</sup> coordination-driven self-assembling of  
amorphous amphiphilic aromatic block copolymer  
into semi-crystallized nanostructures with  
enhanced fluorescence emission

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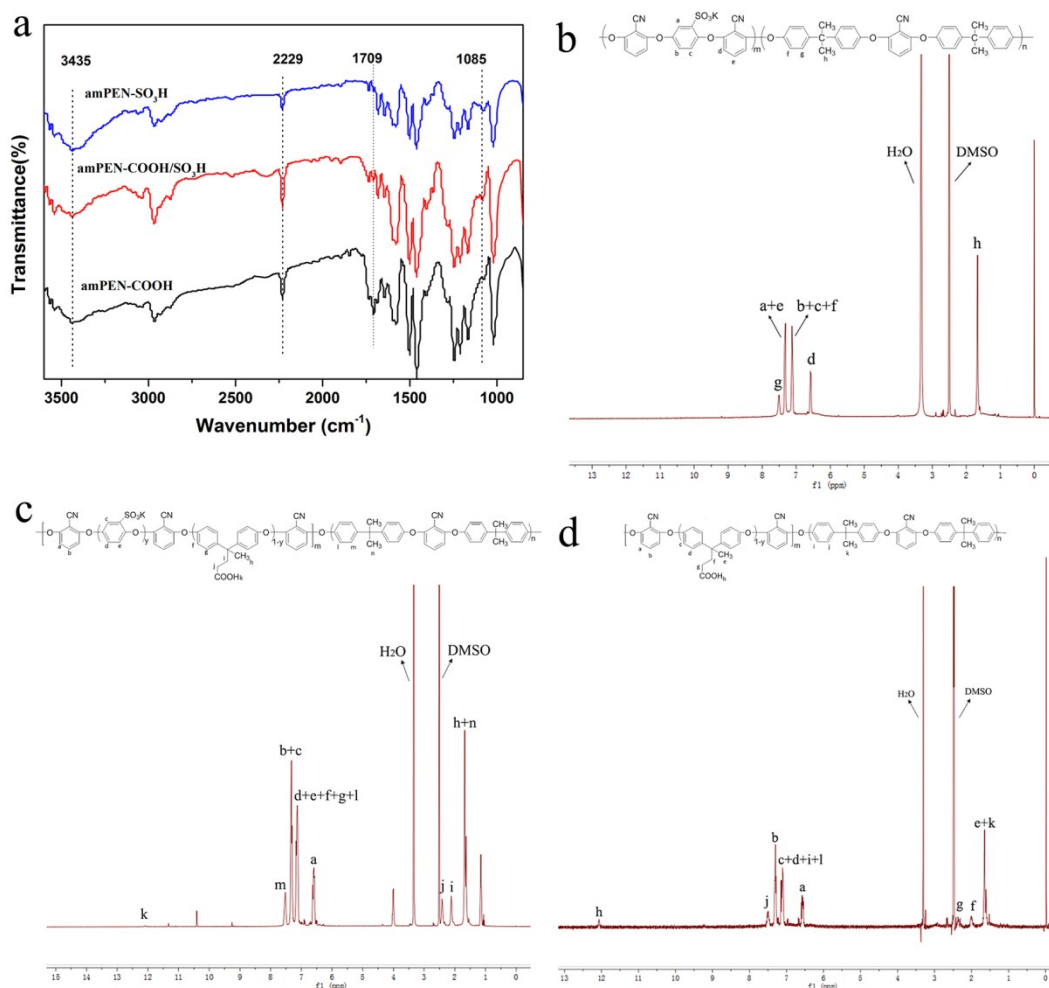
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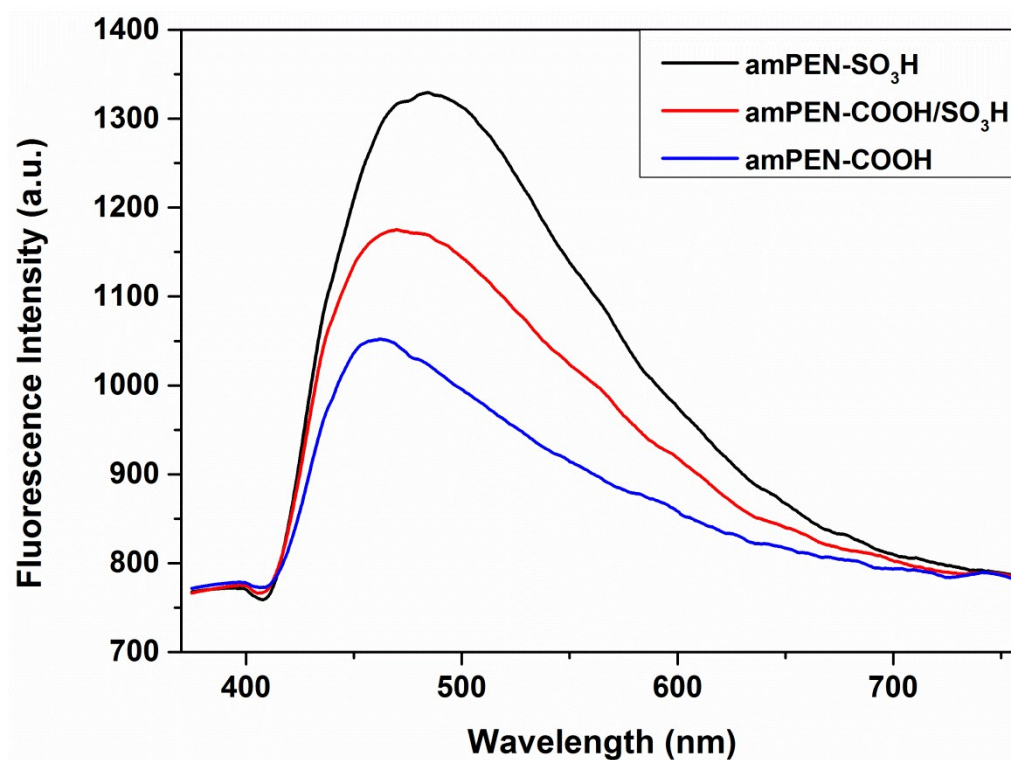
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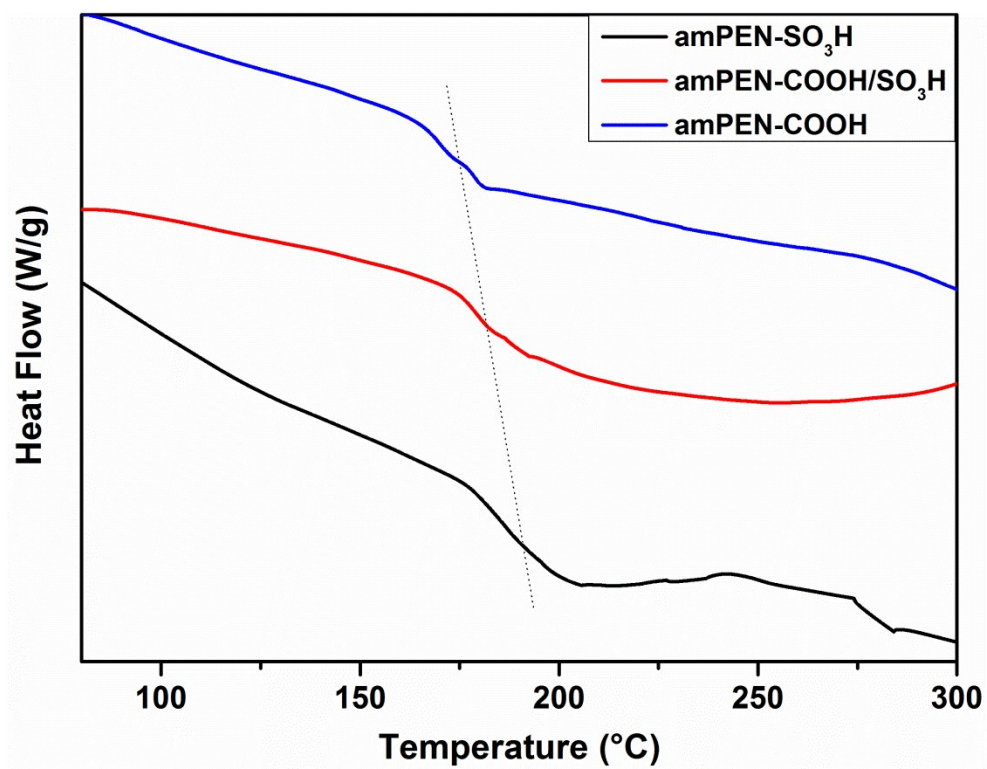
As shown in Fig. S1a, we can see the characteristic band of  $1085\text{ cm}^{-1}$  corresponding to the asymmetric stretching vibrations of aromatic sulfonate. The characteristic band at  $2229\text{ cm}^{-1}$  is attributed to symmetric stretching of nitrile group. The absorption band at  $1709\text{ cm}^{-1}$  is assigned to carboxyl group. Fig. S1b, 1c and 1d show the  $^1\text{H}$  NMR spectra of amPEN-SO<sub>3</sub>H, amPEN-COOH/SO<sub>3</sub>H, amPEN-COOH, respectively. Compared with Fig. S1b, there is a new peak at around 12 ppm both in Fig. S1c and 1d, which is assigned to carboxyl group. The results of FIRT spectra and  $^1\text{H}$  NMR spectra therefore confirmed the targeted copolymers have been synthesized successfully.



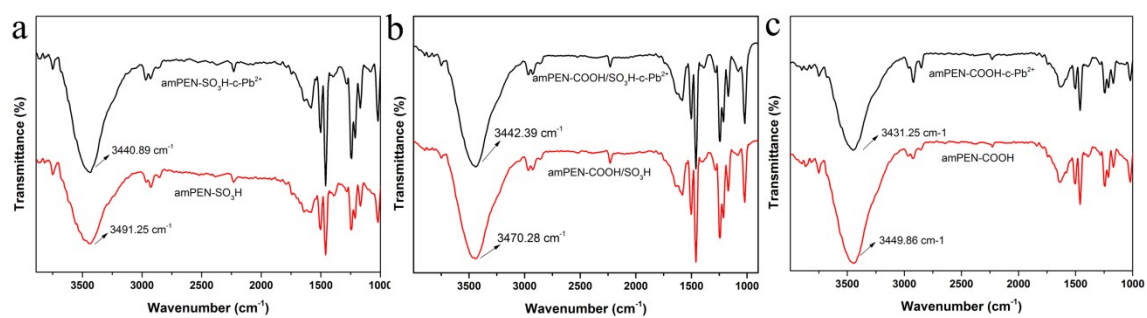
**Fig. S1** The FTIR spectra of synthesized amphiphilic polyarylene ether nitrile (a) and the  $^1\text{H}$  NMR spectra of the synthesized amPEN-SO<sub>3</sub>H (b), amPEN-COOH/SO<sub>3</sub>H (c), amPEN-COOH (d).



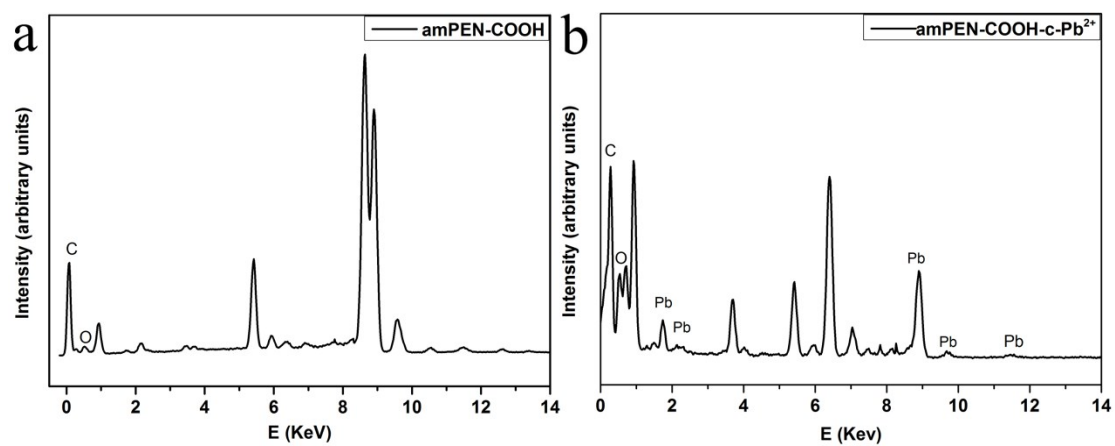
**Fig. S2** The fluorescence emission spectra of the synthesized amPEN-SO<sub>3</sub>H, amPEN-COOH/SO<sub>3</sub>H and amPEN-COOH in DMF solution with the concentration of 2.5 mg mL<sup>-1</sup>.



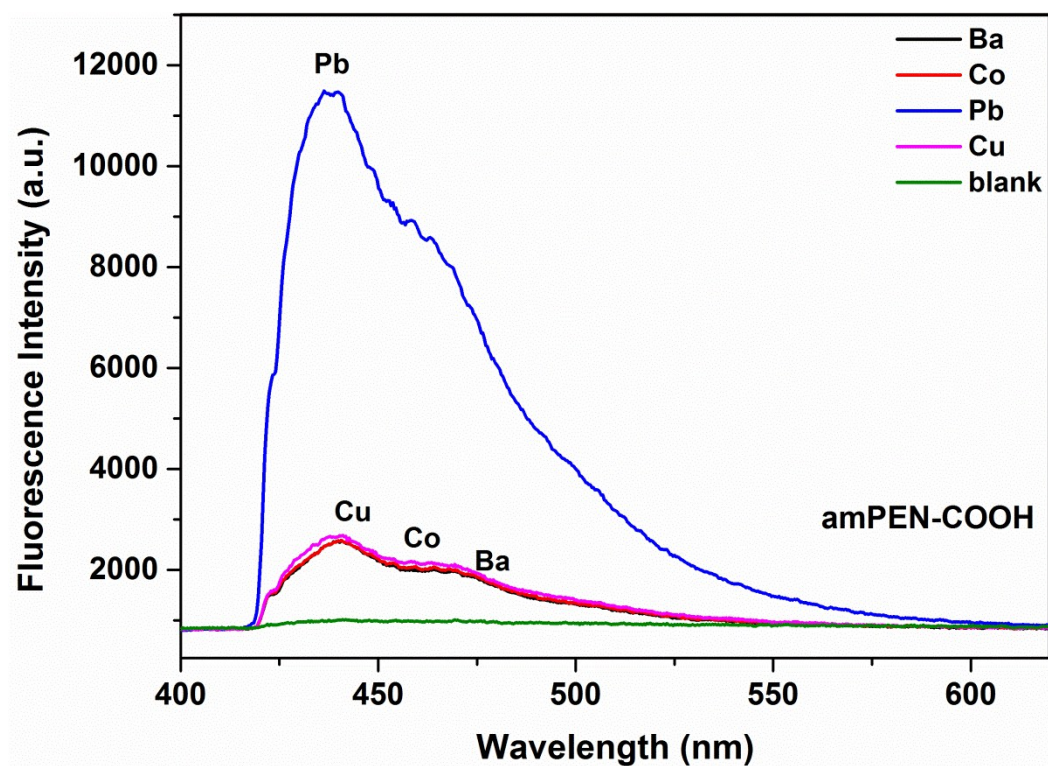
**Fig. S3** The DSC curves of the prepared amPEN-SO<sub>3</sub>H, amPEN-COOH/SO<sub>3</sub>H and amPEN-COOH.



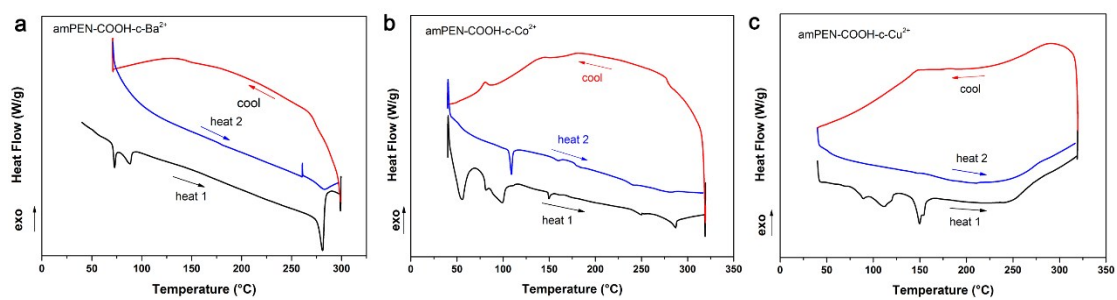
**Fig. S4** The FI-IR spectra of amPEN nanospheres and Pb<sup>2+</sup> coordinated amPEN nanospheres.



**Fig. S5** The EDS spectra of amPEN nanospheres and amPEN-COOH-c-Pb<sup>2+</sup> nanospheres.



**Fig. S6** The fluorescence emission spectra of the different divalent metal ions coordinated amPEN-COOH nanospheres.



**Fig.S7** The DSC curves of polymer nanostructures obtained via the self-assembly of amPEN-COOH in the presence of Ba<sup>2+</sup> (a), Co<sup>2+</sup> (b) and Cu<sup>2+</sup> (c), respectively.