

Supporting Information for:

Oxidation- and thermo-responsive poly(N-isopropylacrylamide-co-2-hydroxyethyl acrylate) hydrogels cross-linked via diselenides for controlled drug delivery

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Table S1

| Samples | Feed compositions / mmol | | | Theoretical NIPAM content (mol%) | NIPAM content in the copolymer (mol%) ^a | Yield (%) |
|---------|--------------------------|-------|------|----------------------------------|--|-----------|
| | NIPA M | HEA | AIBN | | | |
| PNH9/1 | 45 | 5.00 | 3.40 | 90 | 93 | 93 |
| PNH | 45 | 11.25 | 3.40 | 80 | 84 | 93 |
| PNH5/5 | 45 | 45.00 | 3.40 | 50 | 62 | 95 |

^a calculated from ¹H NMR analysis results.

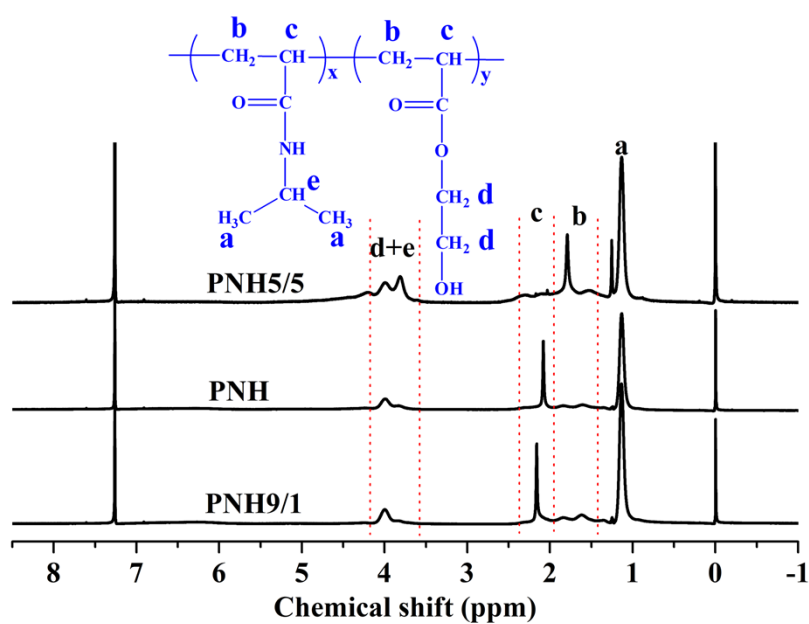
Table S1. Feed and result compositions of poly(NIPAM-co-HEA) copolymers.**Fig. S1****Fig. S1.** ¹H NMR spectra of PNH9/1, PNH and PNH5/5 in CDCl₃.

Fig. S2

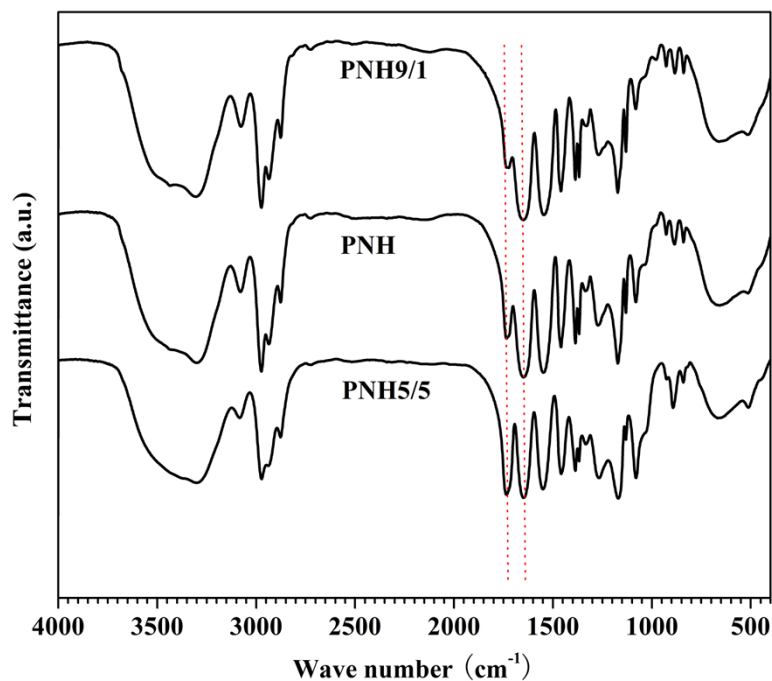


Fig. S2. FT-IR spectra of PNH9/1, PNH and PNH5/5.

Table S2

| <i>Sample</i> | <i>Compositions (g)</i> | | <i>Gelation time (h)^a</i> | <i>Yield (%)</i> |
|-----------------|-------------------------|----------------|--------------------------------------|------------------|
| | PNH | IPDI-SeSe-IPDI | | |
| PNH-SeSe1 | 3.28 | 0.20 | No gelation | – |
| PNH-SeSe2/Gel 1 | 3.28 | 0.60 | 6 | 86 |
| PNH-SeSe3/Gel 2 | 3.28 | 1.80 | 3 | 95 |

^aThe gelation time was determined at the time point when the mixture solution became an immobile gel in the inverted cuvette at room temperature.

Table S2. Compositions, gelation time and yields of the mixing solution containing PNH and IPDI-SeSe-IPDI.

Fig. S3

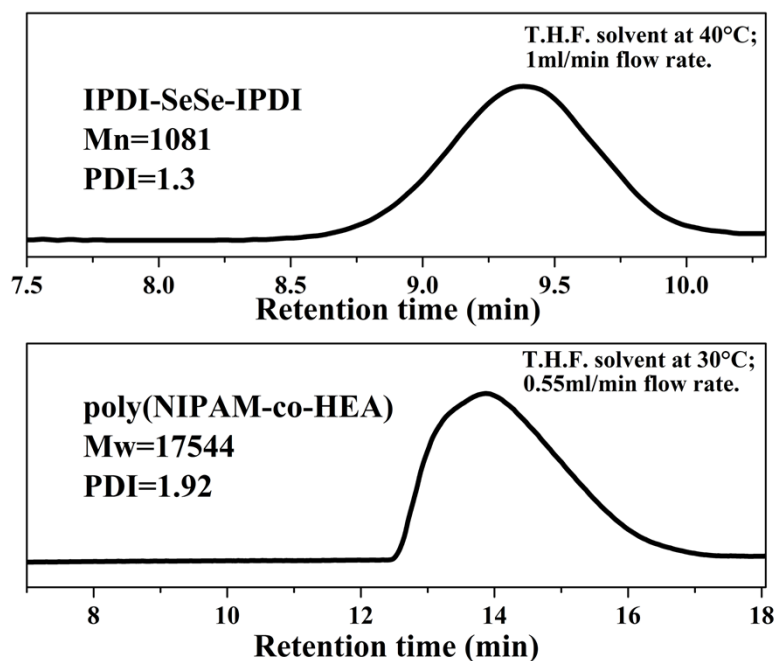


Fig. S3. GPC trace of copolymer PNH and the crosslinking agent IPDI-SeSe-IPDI.

Fig. S4.

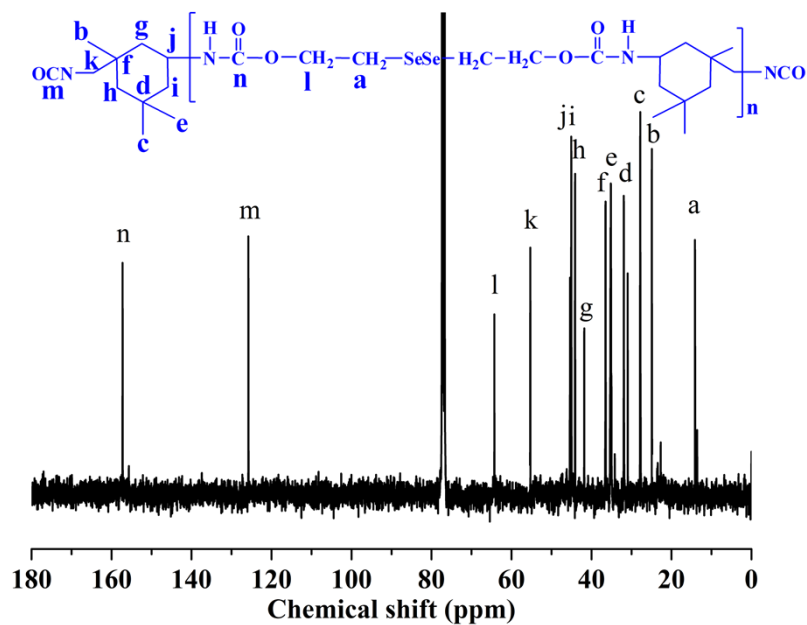


Fig. S4. ^{13}C NMR spectrum of the crosslinker IPDI-SeSe-IPDI in CDCl_3 .

Fig. S5.

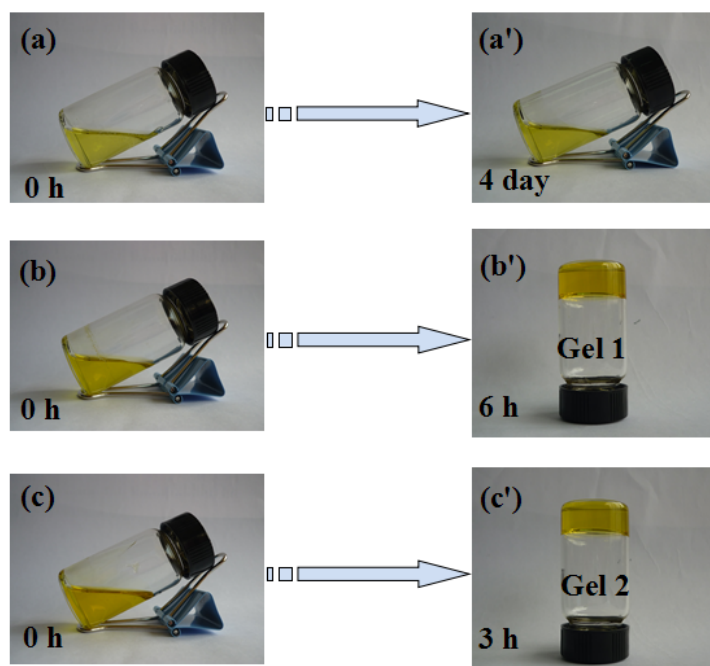


Fig. S5. Time-dependent sol-gel transition of mixing solution containing PNH copolymers (3.28 g) and IPDI-SeSe-IPDI crosslinkers varying from 0.20 g (a and a'), 0.60 g (b and b') and 1.80 g (c and c').

Fig. S6.

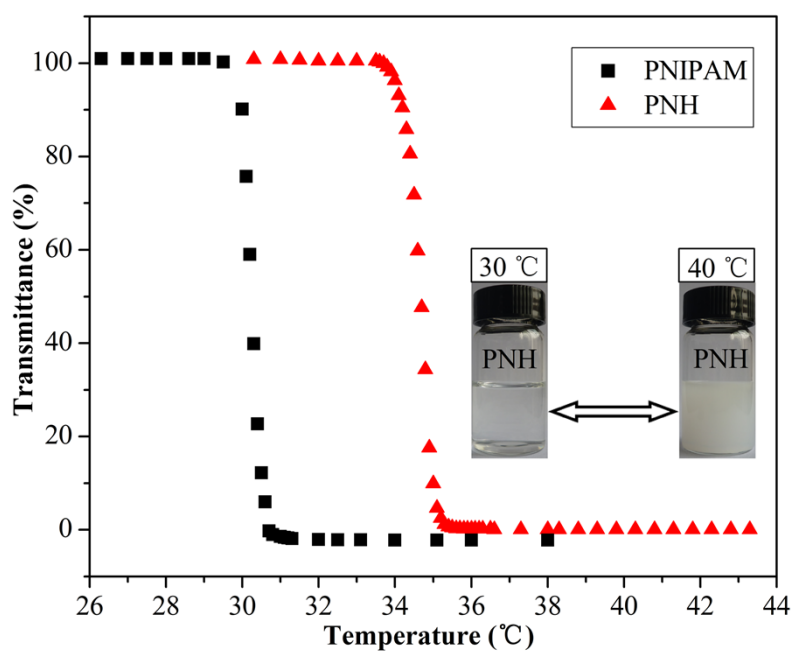


Fig. S6. Turbidity measurements of PNIPAM homopolymer and PNH copolymer.

Fig. S7.

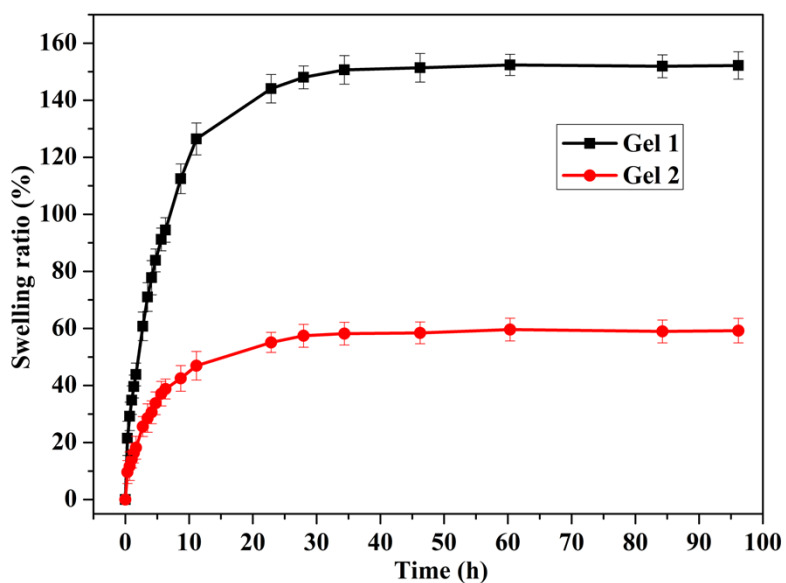


Fig. S7. Dynamic swelling behavior of the PNH-SeSe hydrogels with different crosslinker content (Gel 1, 15% wt; Gel 2, 35% wt) in distilled water at 25 °C.

Fig. S8.

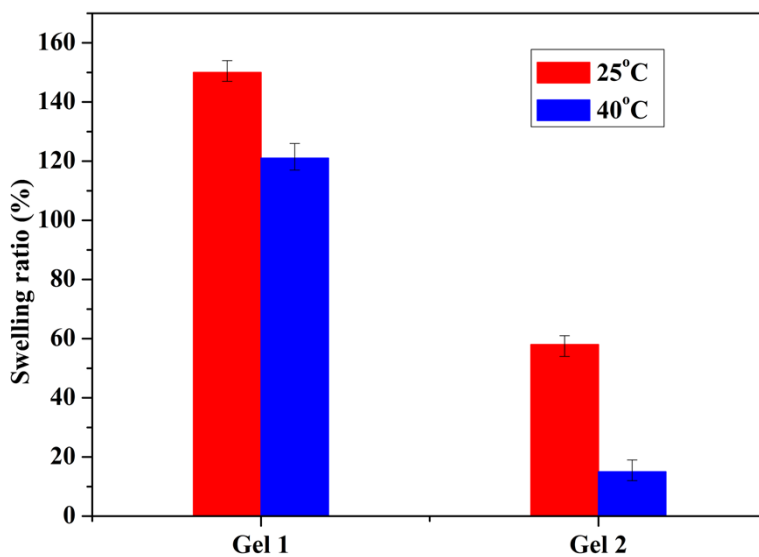


Fig. S8. Thermo-sensitive swelling ratios of the PNH-SeSe hydrogels with different crosslinker content (wt%): Gel 1 (15% wt); Gel 2 (35% wt).

Fig. S9.

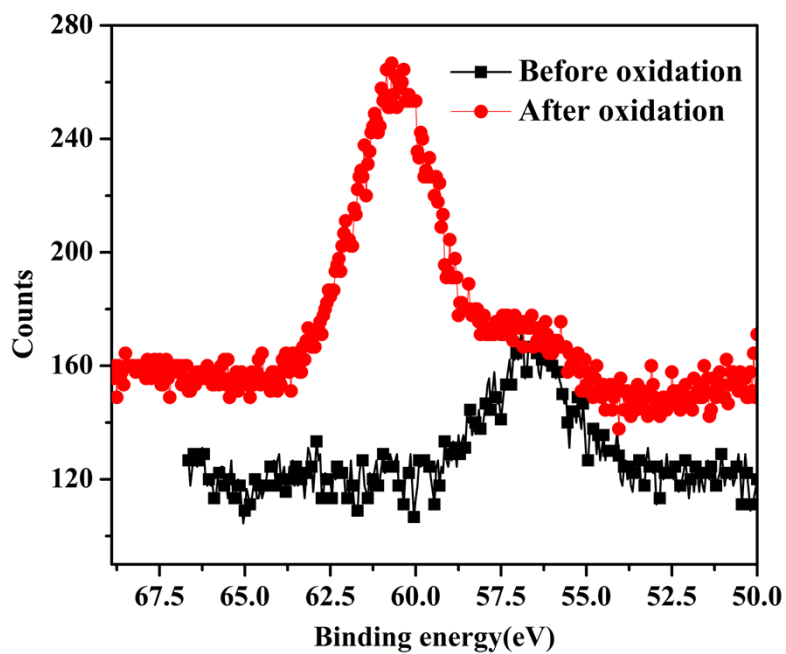


Fig. S9. XPS analysis of the model compound $\text{HOCH}_2\text{CH}_2\text{SeSeCH}_2\text{CH}_2\text{OH}$ before and after oxidation in 0.5%wt H_2O_2 solution.

Fig. S10.

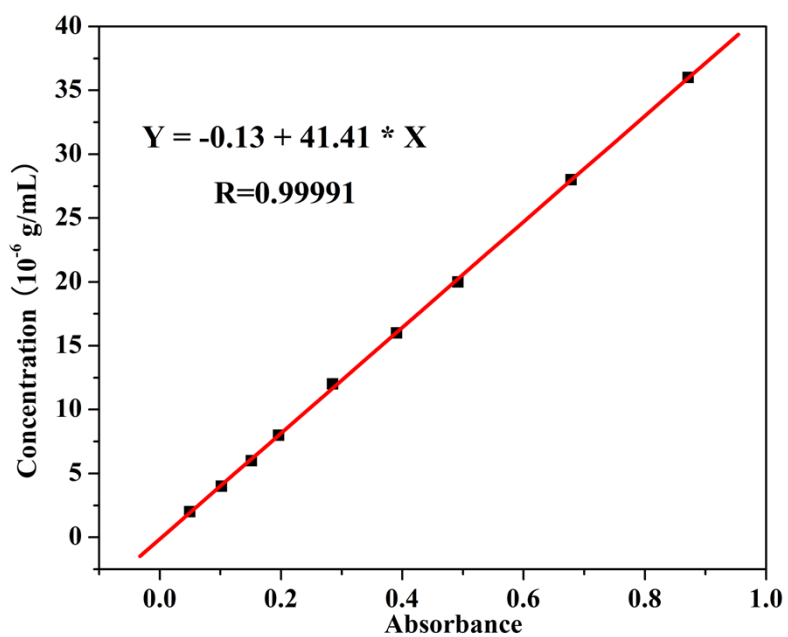


Fig. S10. Calibration curve of the salicylic acid in distilled water.