

**Supporting Information for**

**Creep Resistance in Doubly Crosslinked Dynamic Covalent Networks**

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## Materials and instrumentation

### Materials

The following chemicals were used without purification: chloroform-*d* (CDCl<sub>3</sub>, Cambridge Isotope Laboratories, 99.5%), dimethylsulfoxide-*d*<sub>6</sub> (DMSO-*d*<sub>6</sub>, Cambridge Isotope Laboratories, 99.5%), methanol (MeOH, VWR, 99.8%), *tert*-butyl acetoacetate (VWR, ≥ 95.0%), 1,1,1-trimethylolpropane (Sigma-Aldrich, 99%), trifluoroacetic acid (TFA, Sigma-Aldrich, 99%), hexamethylenediamine (Sigma-Aldrich, 98%), xylenes (Sigma-Aldrich, 75%), hexanes (VWR, 99.5%), ethyl acetate (EtOAc, VWR, 99.5%), dichloromethane (DCM, VWR, 99.5%). Visualization of spot-on pre-coated silica gel G254 TLC plates (Merck) was achieved using the UV light (254 nm), KMnO<sub>4</sub> test, and ninhydrin test. Synthesized compounds were purified on silica gel column chromatographic using **100-200** mesh Silica gel.

### Instrumentation

#### Nuclear Magnetic Resonance Spectroscopy (<sup>1</sup>H NMR)

<sup>1</sup>H, <sup>13</sup>C, and DEPT-135 spectra were recorded using a Bruker 400 MHz and Bruker AV-400 MHz NMR Spectrometer. <sup>1</sup>H NMR and <sup>13</sup>C NMR chemical shifts are expressed in parts per million (ppm, δ scale). CDCl<sub>3</sub> was referenced to CHCl<sub>3</sub> at δ 7.26 ppm in <sup>1</sup>H NMR and δ 77 ppm in <sup>13</sup>C NMR. DMSO-*d*<sub>6</sub> was referenced to CHD<sub>2</sub>SOCD<sub>3</sub> at δ 2.50 ppm in <sup>1</sup>H NMR and δ 39 ppm in <sup>13</sup>C NMR. D<sub>2</sub>O was referenced to H<sub>2</sub>O at δ 4.79 ppm in <sup>1</sup>H NMR. Multiplicities are reported as follows: s, singlet; d, doublet; t, triplet; q, quartet; m, multiplet.

#### Oscillatory Shear Rheology

Oscillatory shear rheology was performed on a TA Instruments Discovery Hybrid Rheometer (DHR-2) with a 20 mm parallel plate geometry and a gap size of 1 mm. Frequency sweeps were performed at 0.03% strain at 120, 130, 140, and 150 °C. Creep recovery experiments were conducted at a stress of 0.01 MPa for 100 seconds and 0 Pa for 100 seconds. All experiments were conducted using a TA Instruments Discovery Hybrid Rheometer (DHR-2) and TA software. Stress relaxation experiments were conducted at 0.3% strain at the desired temperatures.

#### Dynamic Mechanical Analysis (DMA)

DMA experiments were collected on a TA Q800 DMA (TA Instruments, New Castle, DE). DMA experiments provided quantitative information on copolymer viscoelastic and rheological properties by measuring the response of the samples as they were deformed by a 0.05% sinusoidal strain. Using a tensile clamp, each rectangular-shaped sample was heated from room temperature to 180 °C at 3 °C/min. Sample dimensions were kept consistent as length x width x thickness measured approximately (20 mm x 6 mm x 1 mm) for all samples.

All experiments were run at a frequency of 1 Hz. All DMA experiments were recorded using TA's Thermal Advantage for Q Series software.

#### Thermo-gravimetric Analysis (TGA)

TGA experiments were collected on a TA Q5000 TGA, equipped with an autosampler using a 100  $\mu$ L platinum pan. RAMP experiments were heated at 10  $^{\circ}$ C/min from room temperature to 600  $^{\circ}$ C under nitrogen flow (25 mL/min). All TGA experiments were recorded using TA's Thermal Advantage for Q Series software.

#### Differential Scanning Calorimetry (DSC)

DSC experiments were collected on a TA Q1000 DSC equipped with an autosampler and refrigerated cooling system 90, using aluminum hermetic SI 3 sealed pans. RAMP experiments were heated at 10  $^{\circ}$ C/min from -90 to 150  $^{\circ}$ C and cooled at 10  $^{\circ}$ C/min from 150 to -90  $^{\circ}$ C, with 3 min isotherms at each extreme under nitrogen (25 mL/min). All DSC experiments were recorded using TA Thermal Advantage for Q Series software.

#### Melting Point

Melting points were recorded on MEL-TEMP. The heating source is an oil bath.

#### High-Resolution Mass Spectroscopy

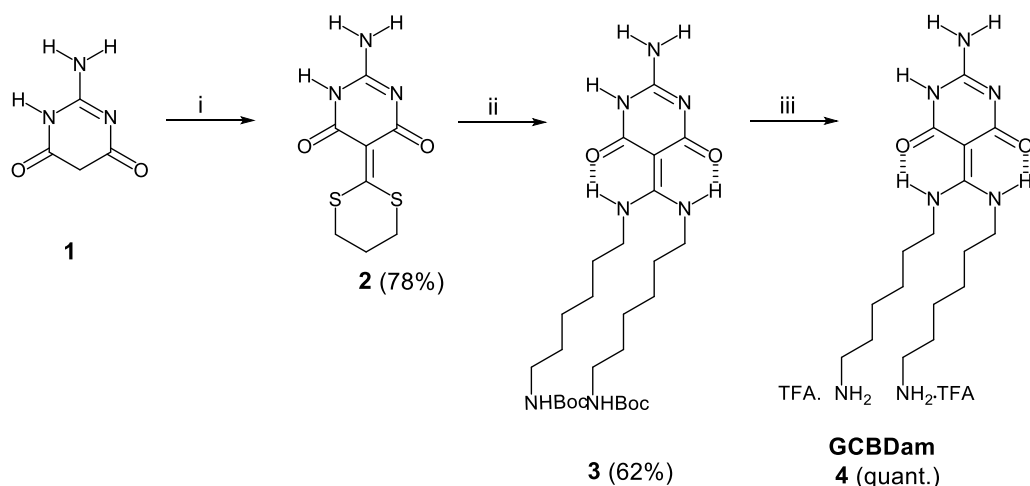
High-resolution mass spectra were recorded on Agilent LC/MSD TOF mass spectrometer by electrospray ionization time-of-flight (ESI-TOF) experiments.

#### FT-IR Spectroscopy

Infrared spectra were collected on a Thermo Nicolet 5700 FT-IR spectrometer equipped with a single bounce diamond stage attenuated total reflectance (ATR) accessory.

## Experimental procedures

### GCBDam synthesis



### Scheme S1. Synthesis of GCBDam.<sup>1</sup>

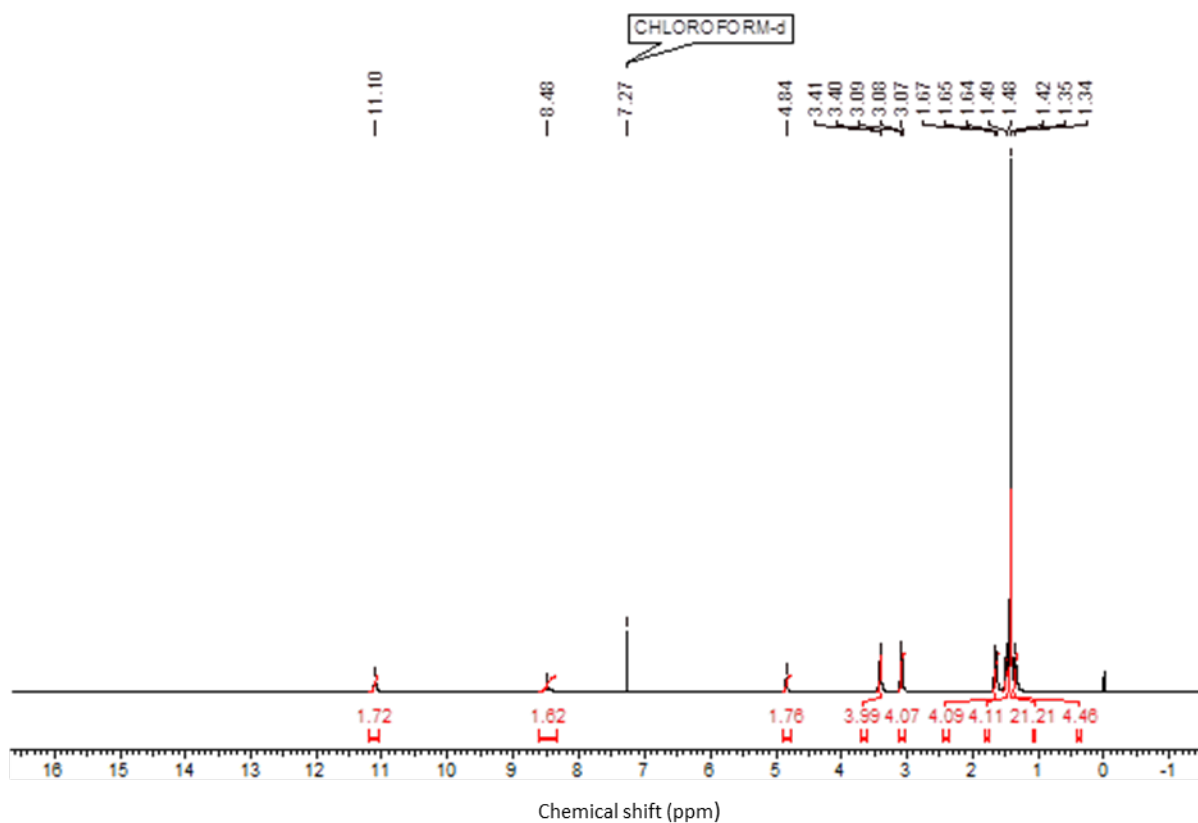
Reaction conditions: i) KOH, CS<sub>2</sub>, 1,3-dibromopropane, H<sub>2</sub>O, rt, 6h; ii) *tert*-butyl (6-aminohexyl)carbamate, DIPEA, DMSO, rt, 5h; iii) TFA:DCM (50:50), 0 °C-rt, 30 min.

Compound 2: To a suspension of **1** (5.00 g, 39.3 mmol) in distilled water (20 mL) at 0 °C, KOH (5.52 g, 98.3 mmol) dissolved in distilled water (10 mL) and CS<sub>2</sub> (8.24 mL, 129 mmol) were added dropwise and the reaction mixture was stirred for 30 min at room temperature. Finally, 1,3-dibromopropane (4.81 mL, 47.2 mmol) was added to the above reaction mixture. The reaction mixture was stirred for 6 h at room temperature and then poured into a citric acid-containing brine solution. The resulting solid was filtered and washed with water followed by diethyl ether and dried in a P<sub>2</sub>O<sub>5</sub> desiccator giving **2** (7.5 g, 78%) as a yellow solid. *Note:* Owing to its poor solubility in common organic solvents, **2** was carried forward for the next reactions without further purification and characterization.

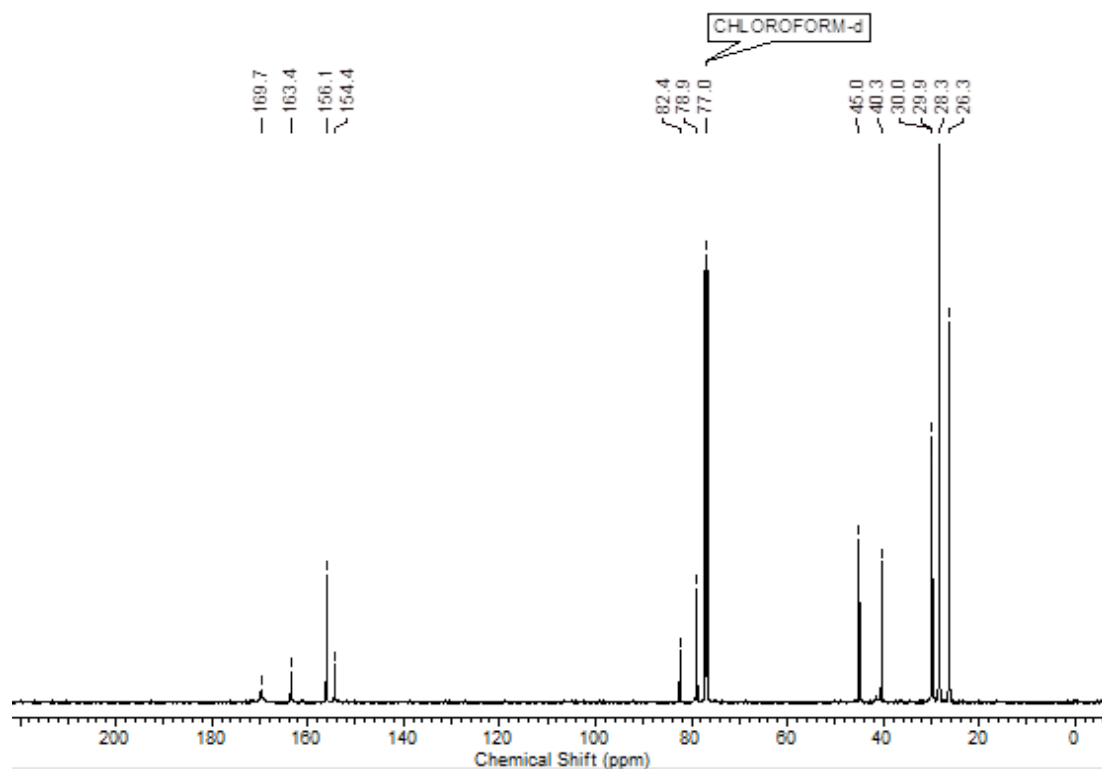
Compound 3: To a suspension of **2** (5.00 g, 20.5 mmol) in DMSO, *tert*-butyl (6-aminohexyl)carbamate (13.8 mL, 61.6 mmol) and DIPEA (10.7 mL, 61.6 mmol) were added and stirred at room temperature for 6 h. After completion of the reaction, the reaction mixture was poured into a citric acid-containing brine solution. The resultant solid was filtered, washed with water, diethyl ether, and dried in a P<sub>2</sub>O<sub>5</sub> desiccator. The residue obtained was purified by column chromatography (eluent: 3% MeOH/DCM, R<sub>f</sub>: 0.5) to afford **3** (7.3 g, 62%) as a white solid: mp: 195-200 °C; <sup>1</sup>H NMR (500 MHz, CDCl<sub>3</sub>) δ: 11.10 (bs, 2H), 8.48 (bs, 2H), 4.84 (bs, 2H), 3.41-3.40 (m, 4H), 3.10-3.07 (m, 4H), 1.67-1.62 (m, 4H), 1.52-1.46 (m, 4H), 1.42 (bs, 21H), 1.37-1.34 (m, 4H); <sup>13</sup>C NMR (125 MHz, CDCl<sub>3</sub>) δ: 169.7, 163.4, 156.1, 154.4, 82.4, 78.9, 45.0, 40.3, 30.0, 29.9, 28.3, 26.3; HRMS (ESI) calculated [M+H]<sup>+</sup> for C<sub>27</sub>H<sub>50</sub>N<sub>7</sub>O<sub>6</sub>: 568.3817, found 568.3809 [M+H]<sup>+</sup>.

Compound 4 (GCBDam): The Boc-protected compound **3** was subjected to deprotection by using TFA: DCM (1:1) mixture for 30 min at 0 °C. After completion of the reaction, the mixture was stripped of solvent and co-

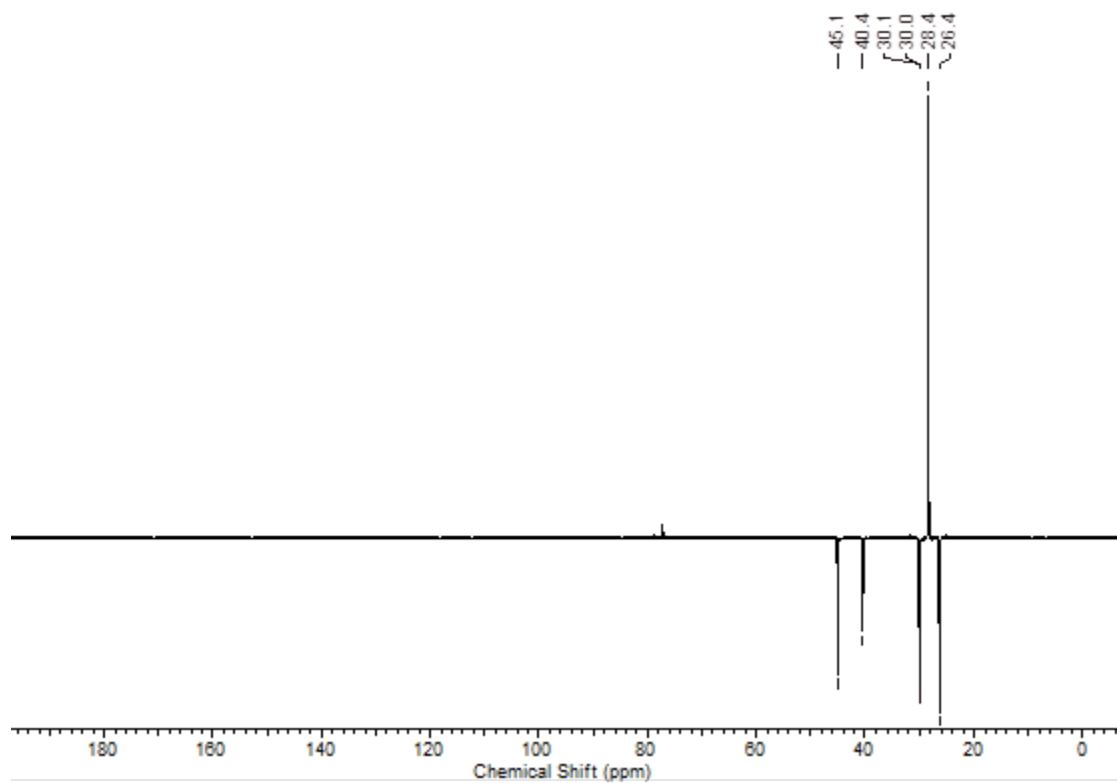
evaporated with DCM (3 times). Further, diethyl ether (Et<sub>2</sub>O) was added to the reaction mixture, and the resultant solid was washed thrice with Et<sub>2</sub>O by decantation and dried under vacuum giving **4**, (**GCBDam**) in quantitative yield, as a white solid: mp: 205-210 °C; <sup>1</sup>H NMR (500 MHz, D<sub>2</sub>O) δ: 3.27-3.26 (m, 4H), 2.94-2.92 (m, 4H), 1.63-1.59 (m, 6H), 1.52 (bs, 2H), 1.38 (bs, 4H), 1.27(bs, 4H); <sup>13</sup>C NMR (125 MHz, D<sub>2</sub>O) δ: 163.3-162.5 (residual TFA signal), 161.5, 158.6, 151.5, 119.8-112.9 (residual TFA signal), 83.2, 44.5, 42.2, 39.3, 28.9, 26.5, 25.5, 25.1; HRMS (ESI) calculated [M+H]<sup>+</sup> for C<sub>17</sub>H<sub>34</sub>N<sub>7</sub>O<sub>2</sub>: 368. 2768, found 368.2758 [M+H]<sup>+</sup>.



**Figure S1.** <sup>1</sup>H NMR spectrum of compound 3.



**Figure S2.**  $^{13}\text{C}$  NMR spectrum of compound 3.



**Figure S3.** DEPT-135 NMR spectrum of compound 3.

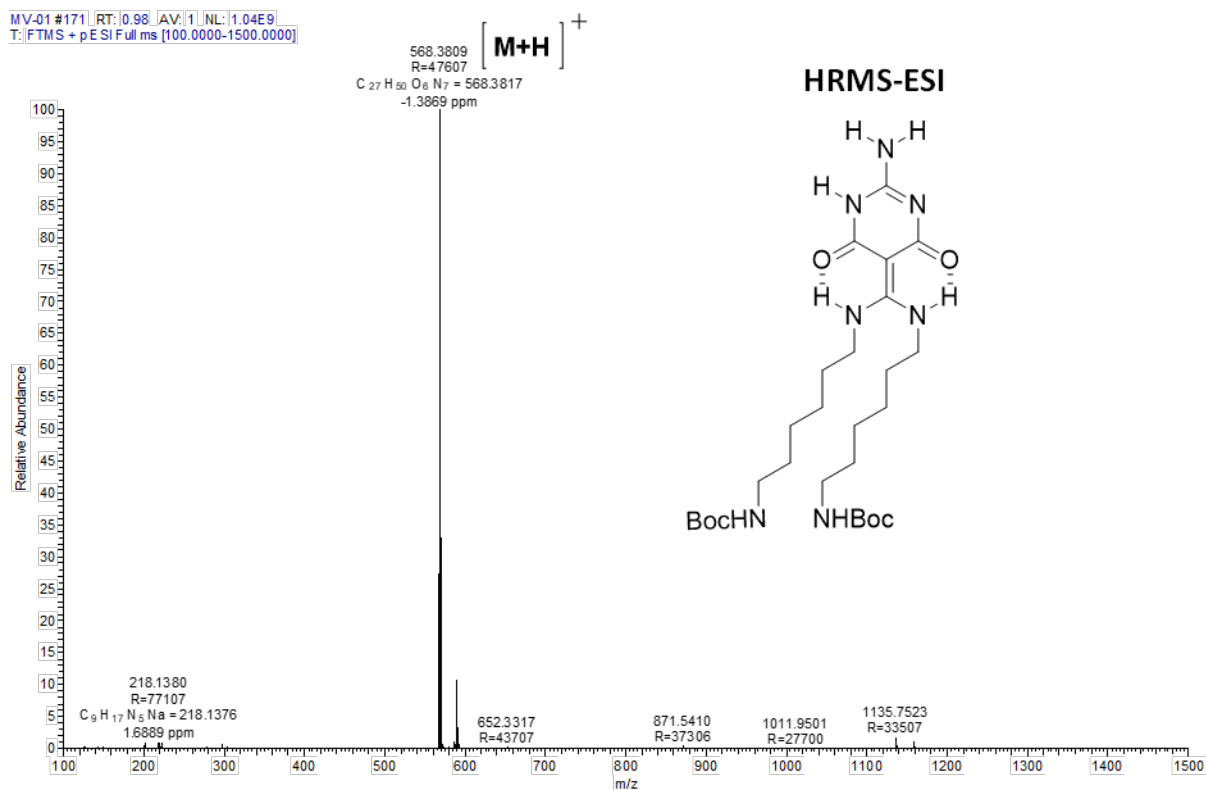


Figure S4. HRMS-ESI of compound 3.

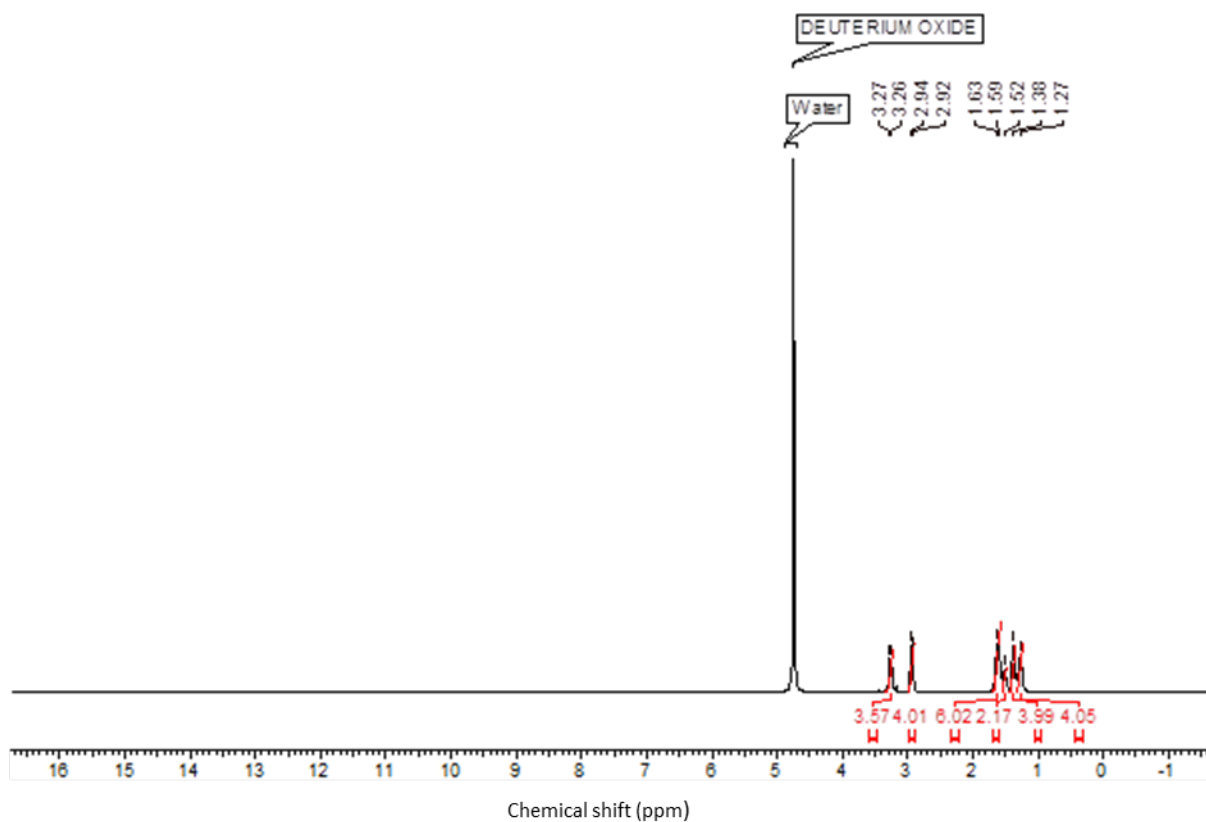


Figure S5. <sup>1</sup>H NMR spectrum of GCBDam.



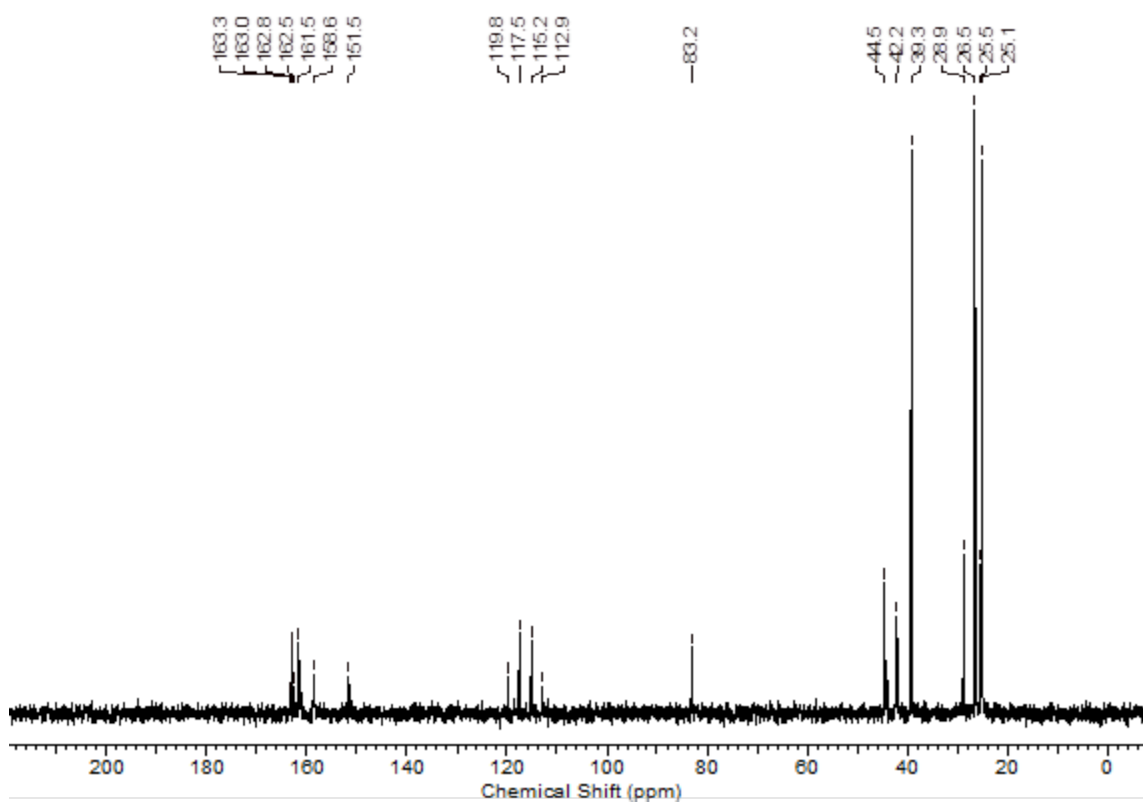


Figure S6.  $^{13}\text{C}$  NMR spectrum of GCBDam.

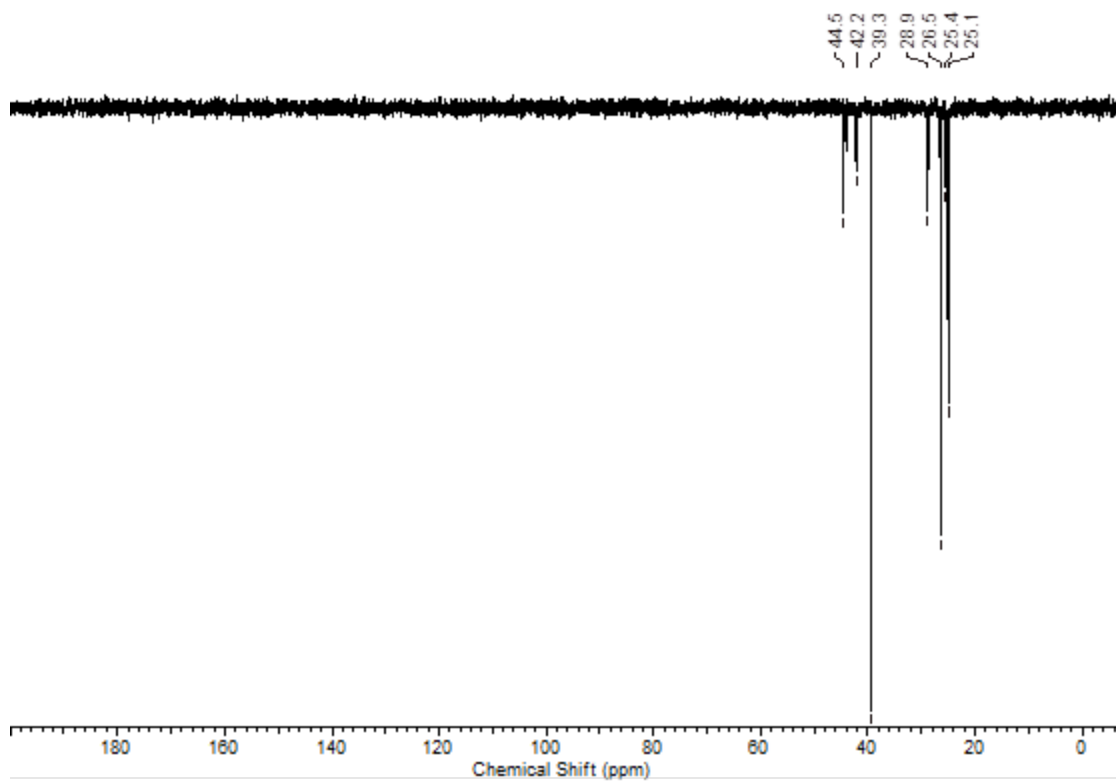
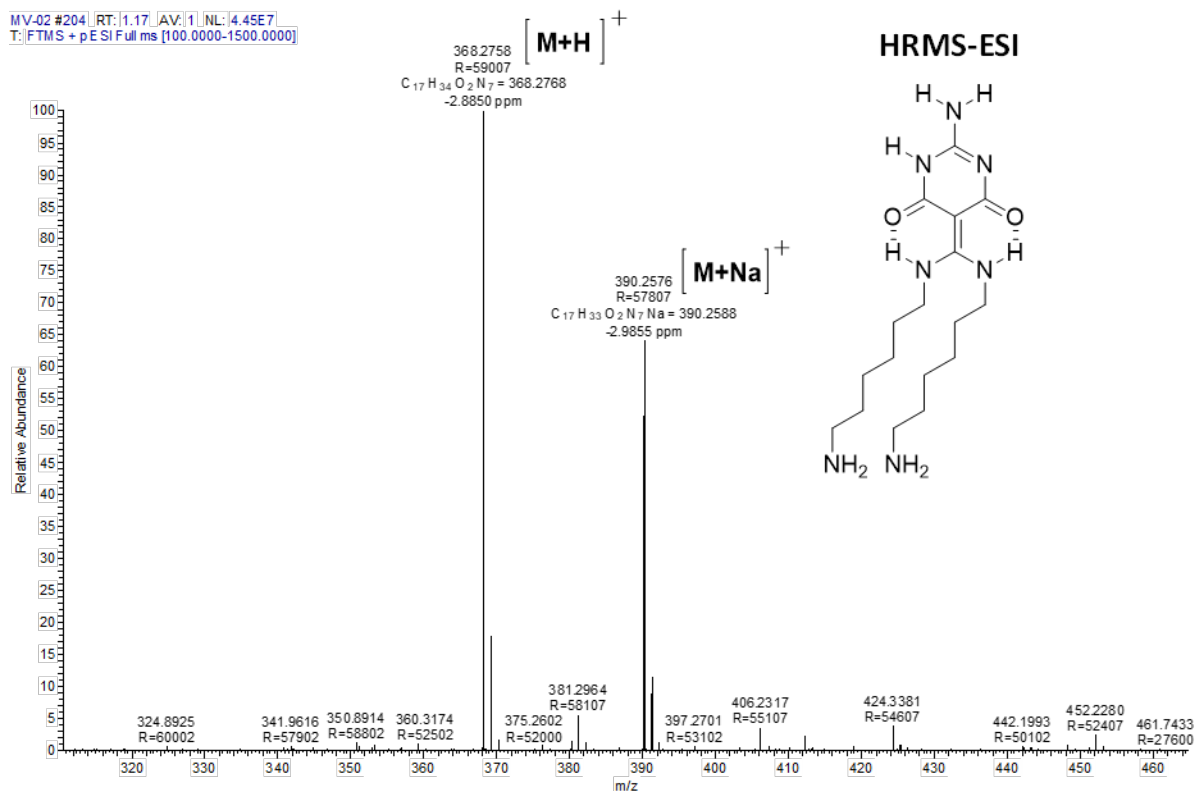
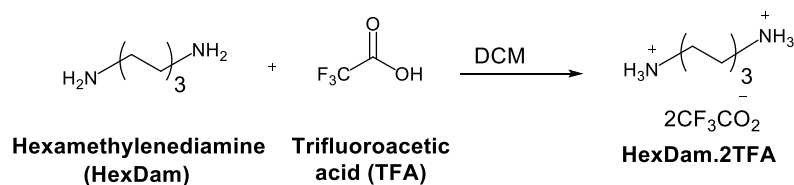


Figure S7. DEPT-135 NMR spectrum of GCBDam.



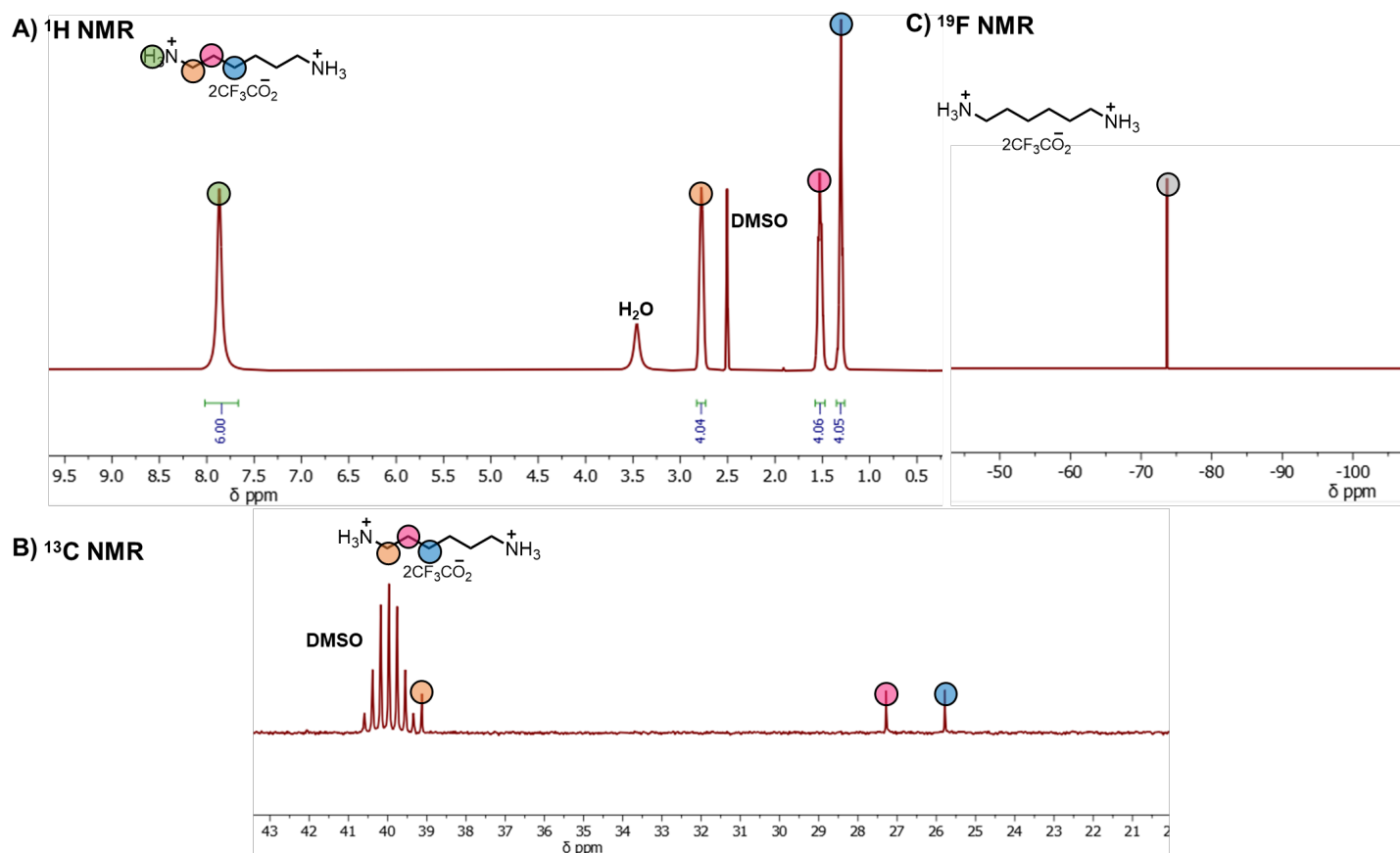
**Figure S8.** HRMS-ESI of GCBDam.

Trifluoroacetic acid salt of hexamethylenediamine synthesis (HexDam.2TFA)



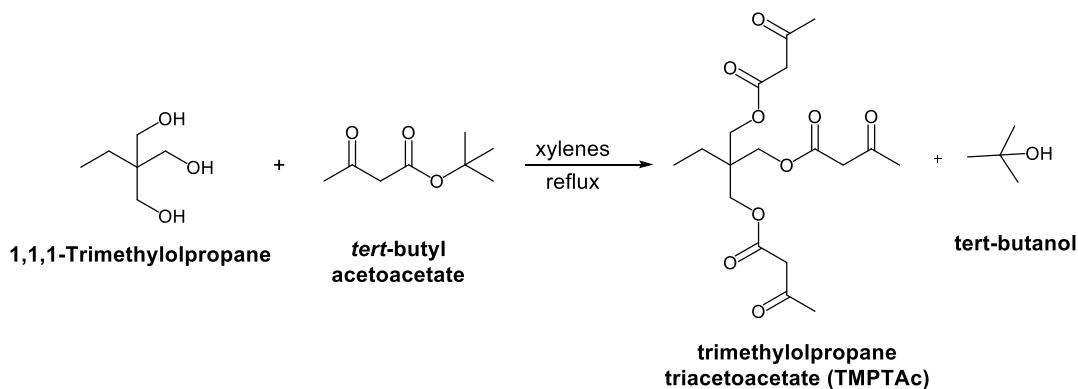
**Scheme S2.** Synthesis of HexDam.2TFA.

Hexamethylenediamine (HexDam) (1.21 g, 10.4 mmol) was dissolved in DCM (4 mL) and excess trifluoroacetic acid (TFA) (1.59 mL, 20.8 mmol) was added dropwise into the solution. The solution was stirred at room temperature for 3 h. The resulting mixture was dried under vacuum using a rotary evaporator to eliminate excess trifluoroacetic acid. Finally, the trifluoroacetic acid salt of hexamethylenediamine was dried in a vacuum oven at 40 °C overnight.



**Figure S9.** A)  $^1\text{H}$  NMR, B)  $^{13}\text{C}$  NMR C)  $^{19}\text{F}$  NMR spectra of HexDam.2TFA.

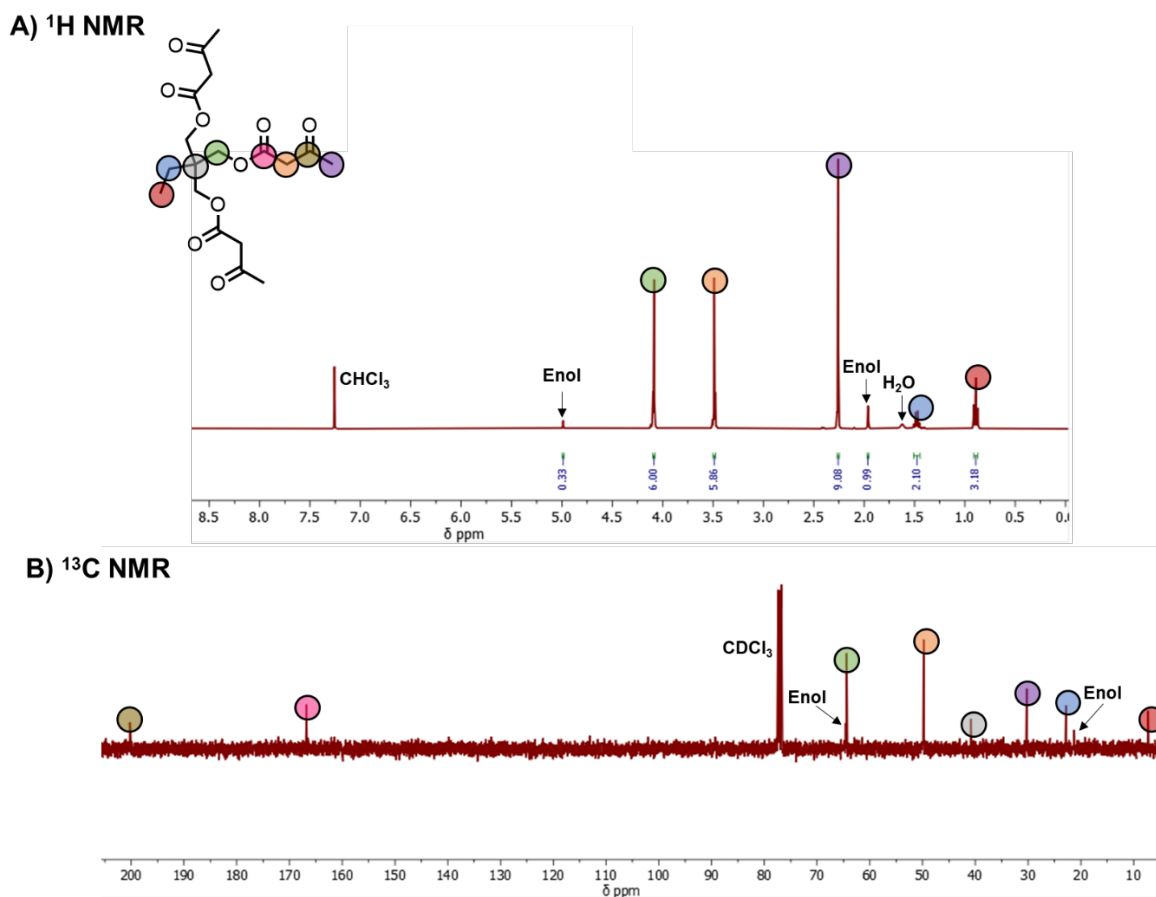
### Crosslinker synthesis



**Scheme S3.** Synthesis of trimethylolpropane triacetoacetate (TMPTAc).

1,1,1-Trimethylolpropane (5.00 g, 37.3 mmol), *tert*-butyl acetoacetate (19.7 mL, 119 mmol), and xylenes (10 mL) were heated overnight in a flask equipped with a distillation column, still head, and thermometers in the base and head of the system.<sup>2</sup> The system was brought to 100 °C under an  $\text{N}_2$  atmosphere, and the *tert*-butanol (t-BuOH) byproduct was collected once the temperature in the still head reached 75-90 °C (bp t-BuOH = 82 °C). The reaction mixture was dried under a vacuum using a rotary evaporator to eliminate trace *tert*-butanol. The

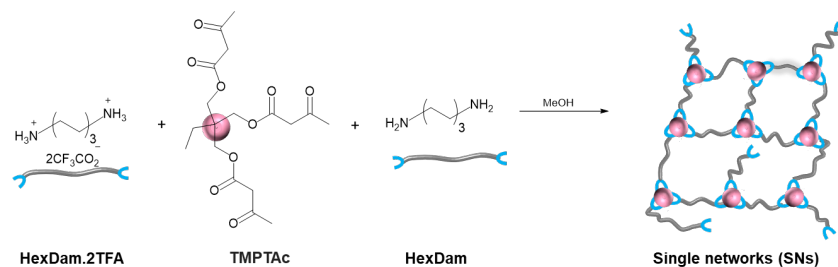
remaining crude product mixture of trimethylolpropane triacetoacetate (TMPTAc) was purified by column chromatography using 1:4 EtOAc and Hexanes.



**Figure S10.** A)  $^1\text{H}$  NMR; B)  $^{13}\text{C}$  NMR spectra of TMPTAc.<sup>3</sup>

## Synthesis of vitrimers

### Single networks (SNs)



#### Scheme S4. Synthesis of SNs.

TMPTAc (0.500 g, 1.29 mmol) was loaded into a petri dish and dissolved in MeOH (3 mL). In separate vials, HexDam (0.286 g, 2.46 mmol) and HexDam.2TFA (0.044 g, 0.130 mmol) were diluted with MeOH (2 mL). The amine solutions were added to the petri dish, and the solution was evaporated at room temperature under an inert atmosphere. The resulting film was further cured at 70-80 °C for up to 4 h under reduced pressure to yield a yellow film. The film was ground up and compression molded at 150 °C for up to 2 h under an inert atmosphere, yielding a transparent material.

### Doubly crosslinked networks (DNs)

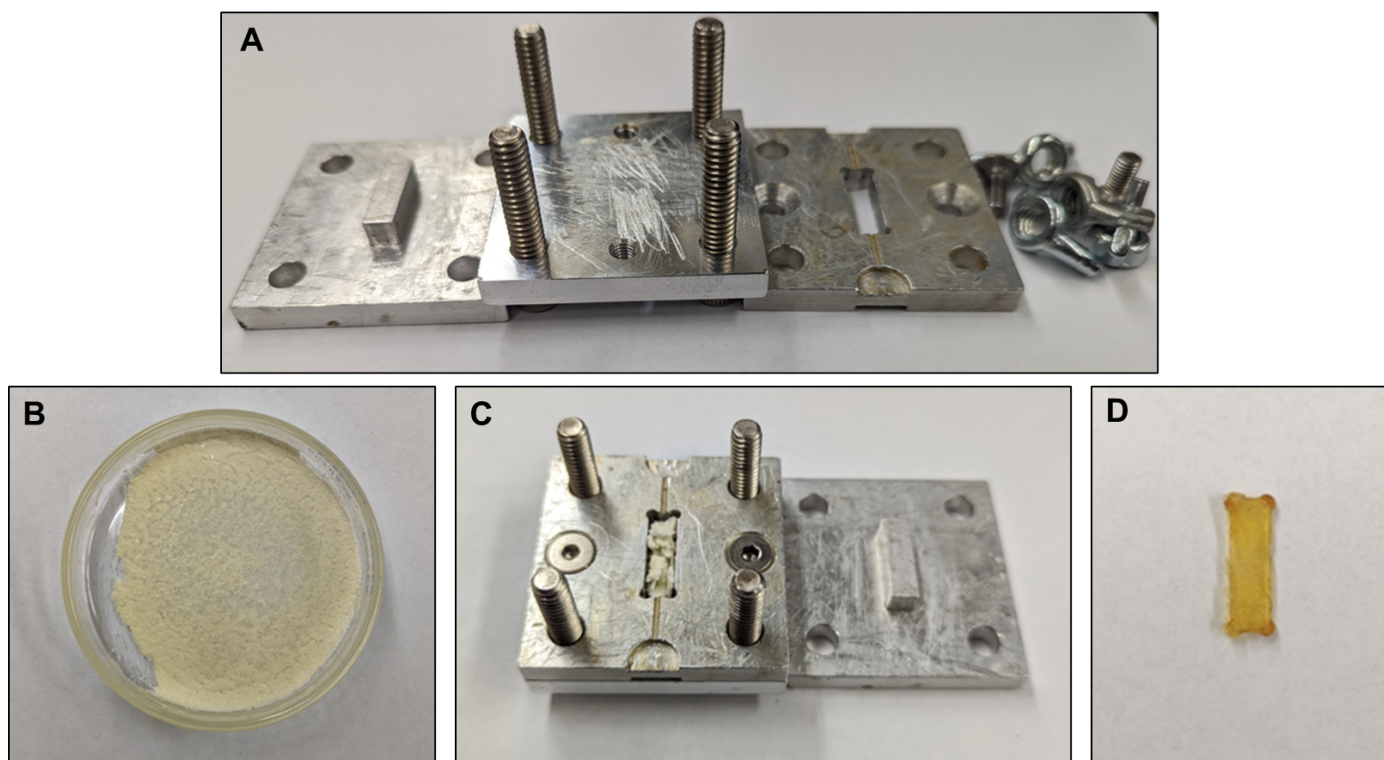
TMPTAc (0.500 g, 1.29 mmol) was loaded into a mold and dissolved in MeOH (3 mL). In separate vials, HexDam (0.286 g, 2.46 mmol) and GCBDam (0.073 g, 0.130 mmol) were diluted with MeOH (2 mL). The amine solutions were added to the mold, and the solution was evaporated at room temperature under an inert atmosphere. The resulting film was further cured at 70-80 °C for up to 4 h under reduced pressure to yield a yellow film. The film was ground up and compression molded at 150 °C for up to 1.5-2 h under an inert atmosphere, yielding a transparent material.

### Gel swelling study

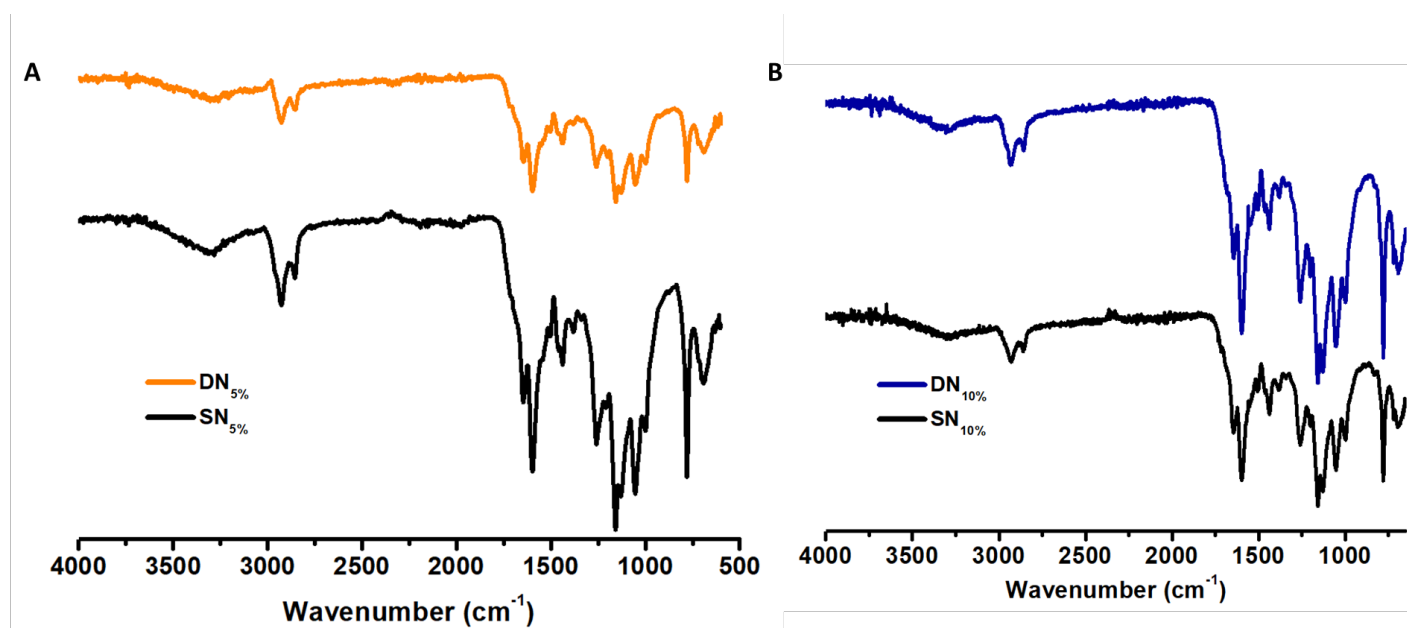
SNs and DNs were heated in THF (50 mg/mL) at 50 °C in a sealed vial 18 h. Final masses were then obtained after gravity filtration, followed by further drying under reduced pressure for 18 h. Recovered mass percentages (i.e., gel fractions) were then calculated for each sample.

**Table S1.** Gel fraction study of materials

Material	Gel fraction
SN <sub>5%</sub>	80.16%
DN <sub>5%</sub>	79.45%
SN <sub>10%</sub>	72.77%
DN <sub>10%</sub>	72.56%

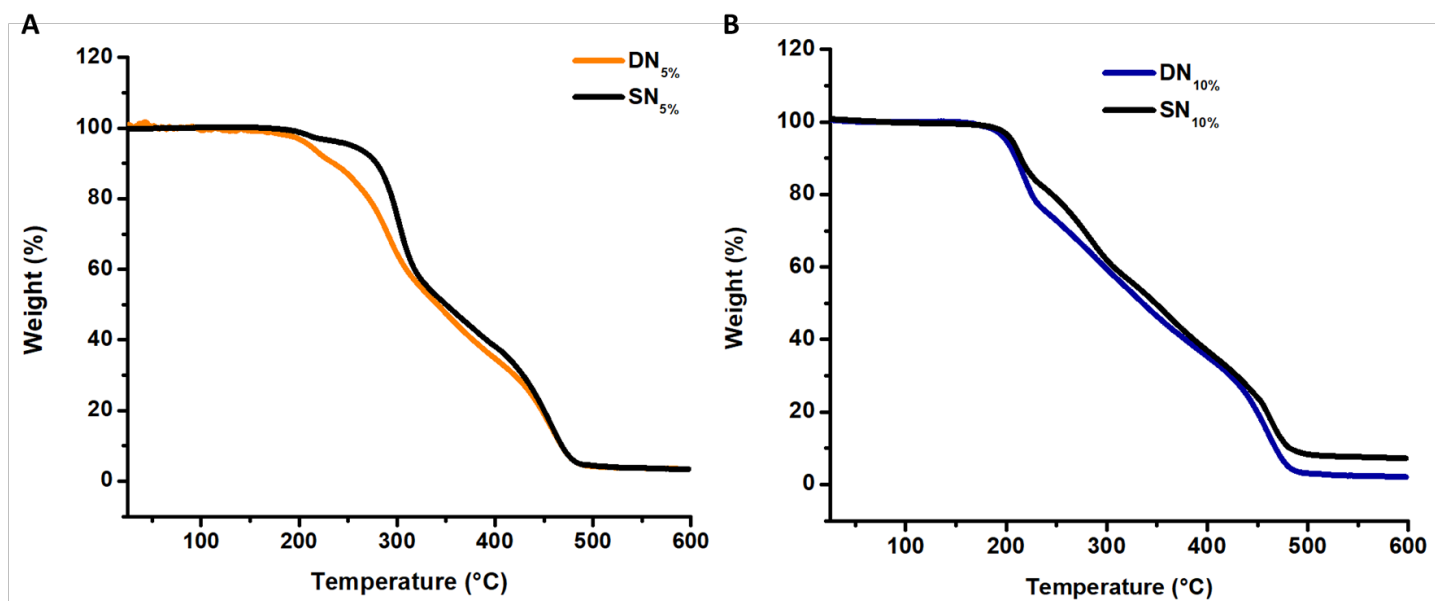


**Figure S11.** A) Molds to prepare rectangular-shaped sample; B) Film formed at 90 °C under vacuum after curing; C) shredded sample in compression mold; D) rectangular-shaped sample for DMA after compression molding.



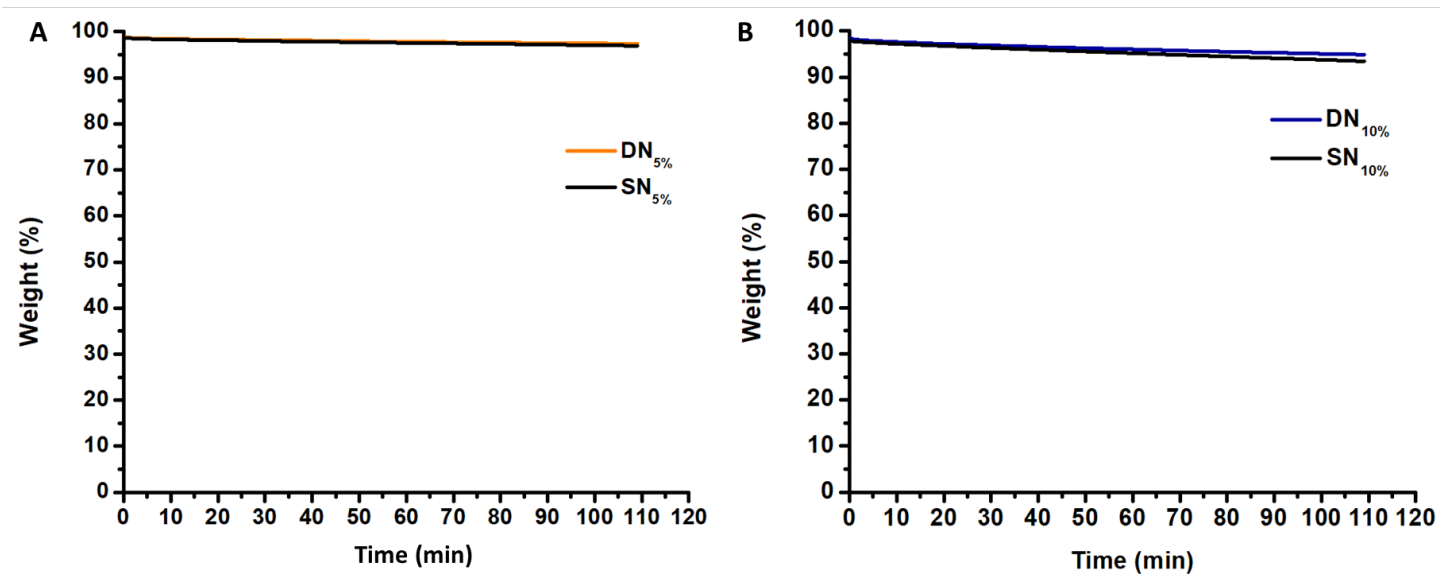
**Figure S12.** IR spectra of A) DN<sub>5%</sub> and SN<sub>5%</sub>; B) DN<sub>10%</sub> and SN<sub>10%</sub>.

## Thermogravimetric analysis (TGA)



**Figure S13.** Comparison of TGA for A) DN<sub>5%</sub> and SN<sub>5%</sub>; B) DN<sub>10%</sub> and SN<sub>10%</sub>.

## Isothermal Study



**Figure S14.** Isothermal TGA analysis at 150 °C for A) DN<sub>5%</sub> and SN<sub>5%</sub>; B) DN<sub>10%</sub> and SN<sub>10%</sub>.

# Rheology experiments

## Frequency sweeps

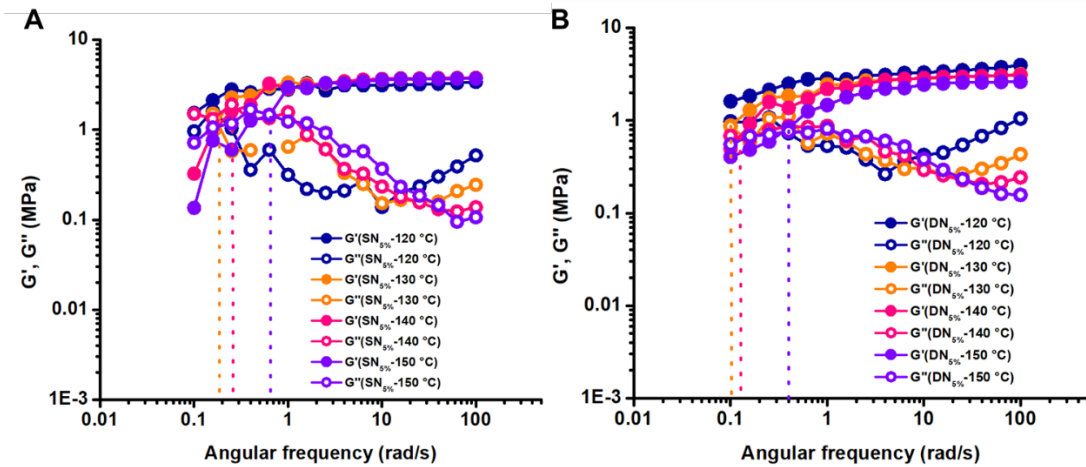


Figure S15. Frequency sweeps of A) SN<sub>5%</sub> and B) DN<sub>5%</sub> over the temperature range 120-150 °C.

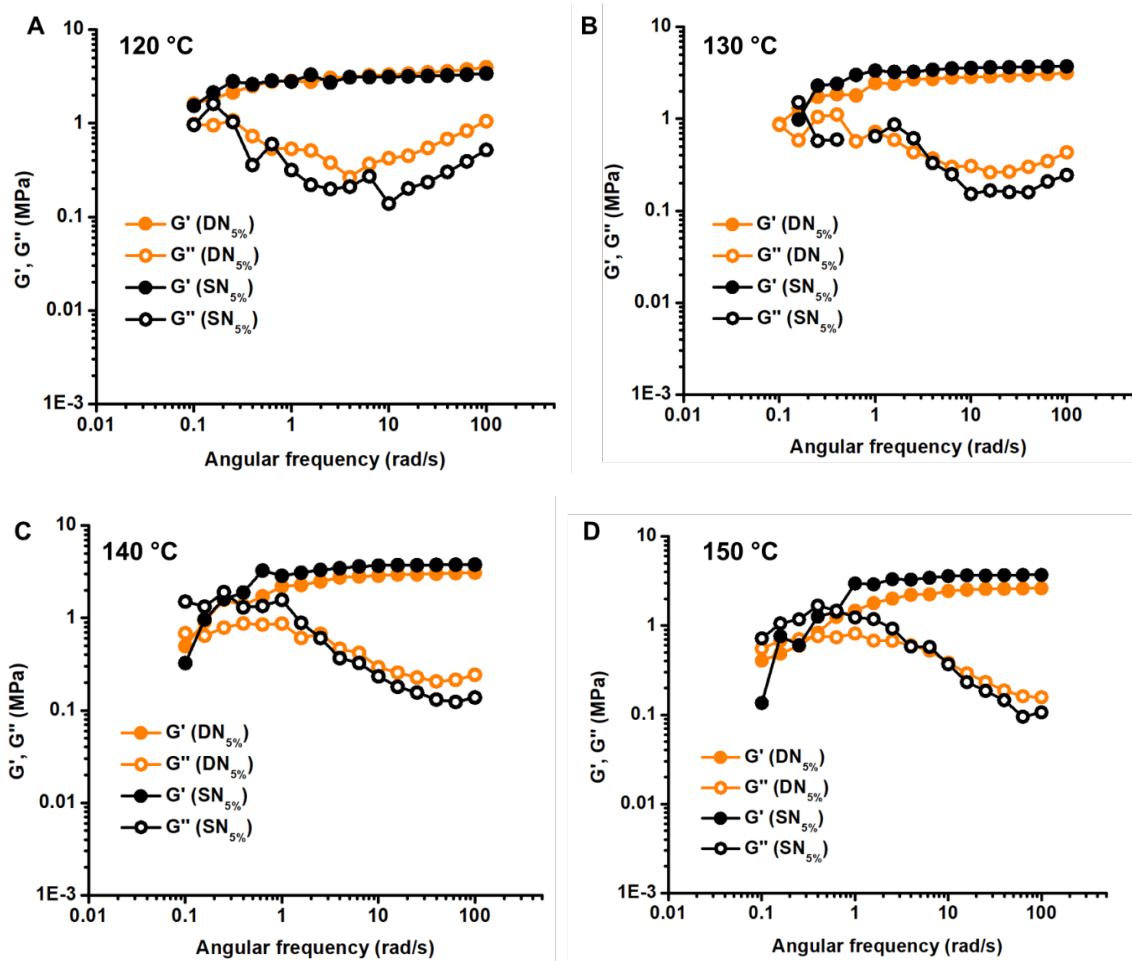


Figure S16. Comparison of the frequency sweeps of SN<sub>5%</sub> and DN<sub>5%</sub> at A) 120 °C; B) 130 °C; C) 140 °C; D) 150 °C.



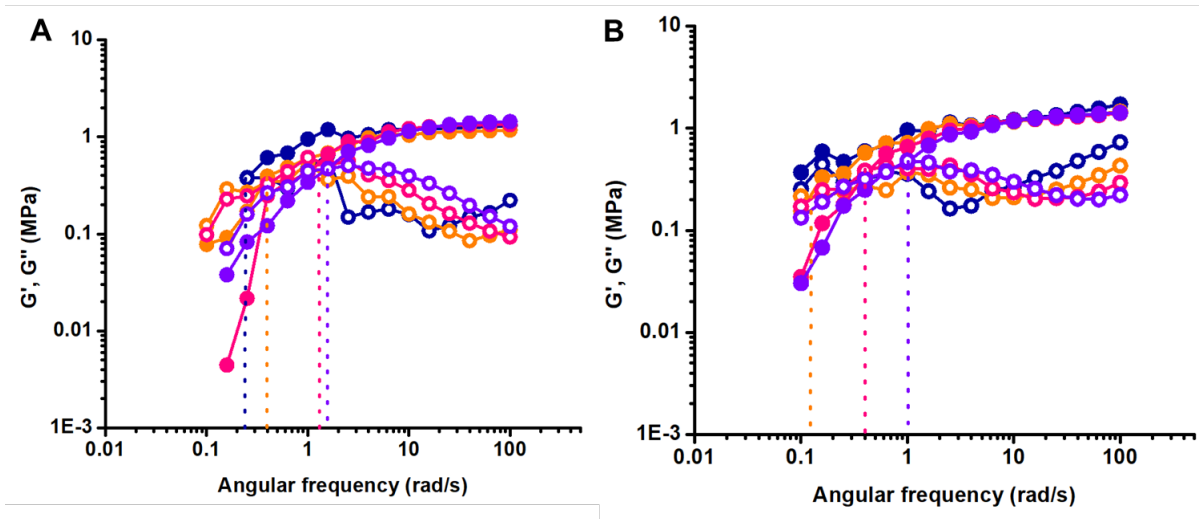


Figure S17. Frequency sweeps of A) SN<sub>10%</sub> and B) DN<sub>10%</sub> over the temperature range 120-150 °C.

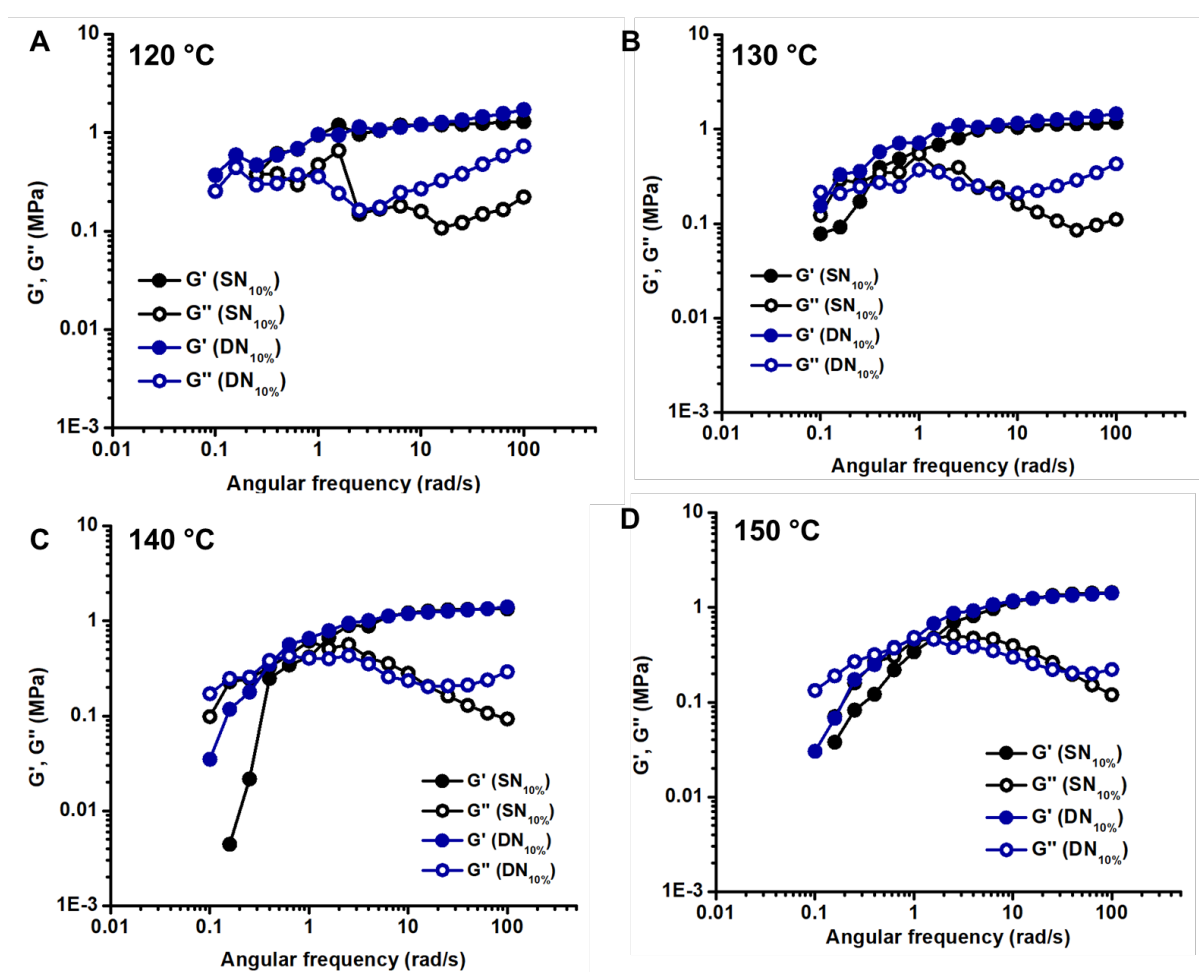
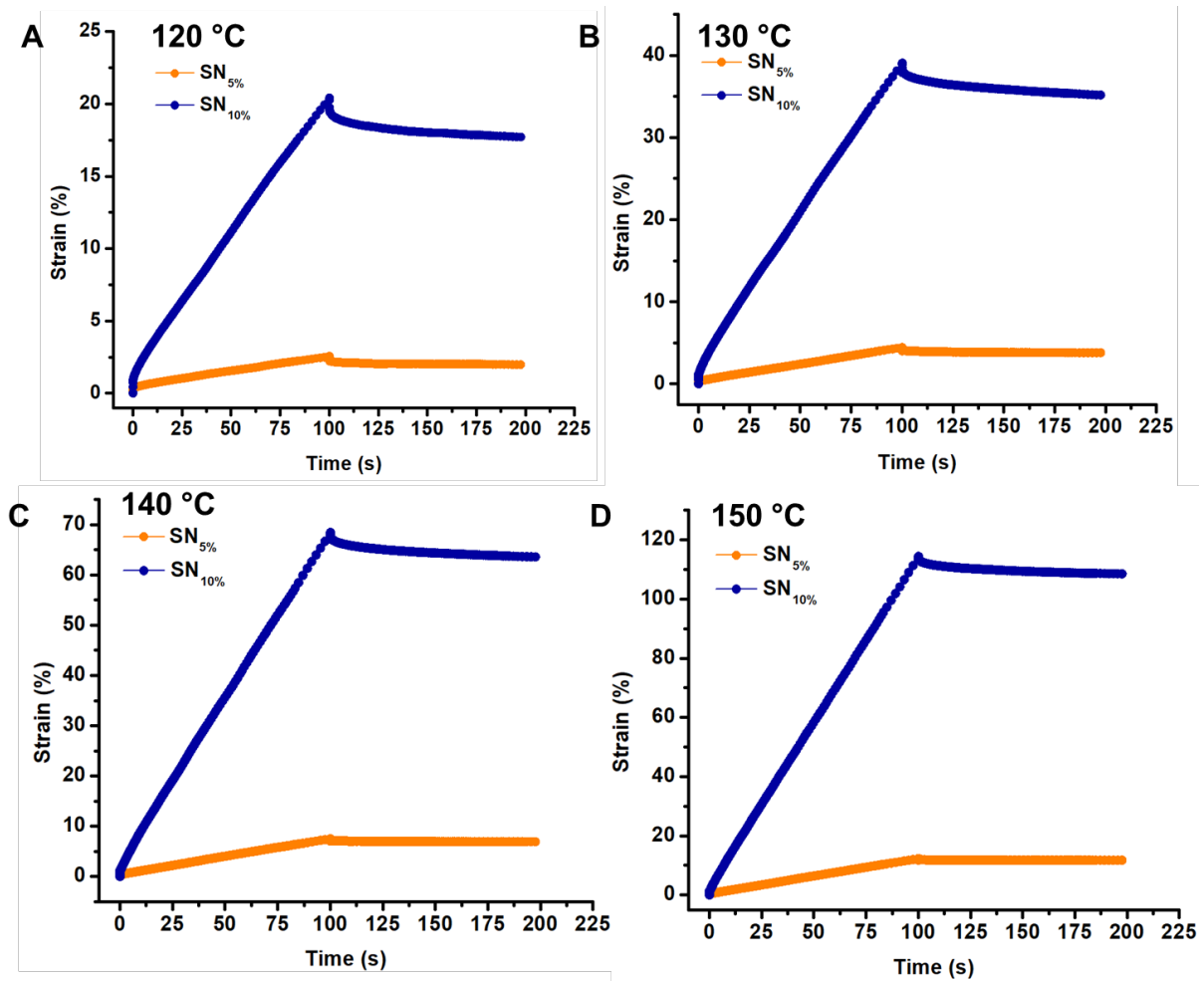


Figure S18. Comparison of the frequency sweeps of SN<sub>10%</sub> and DN<sub>10%</sub> at A) 120 °C; B) 130 °C; C) 140 °C; D) 150 °C.

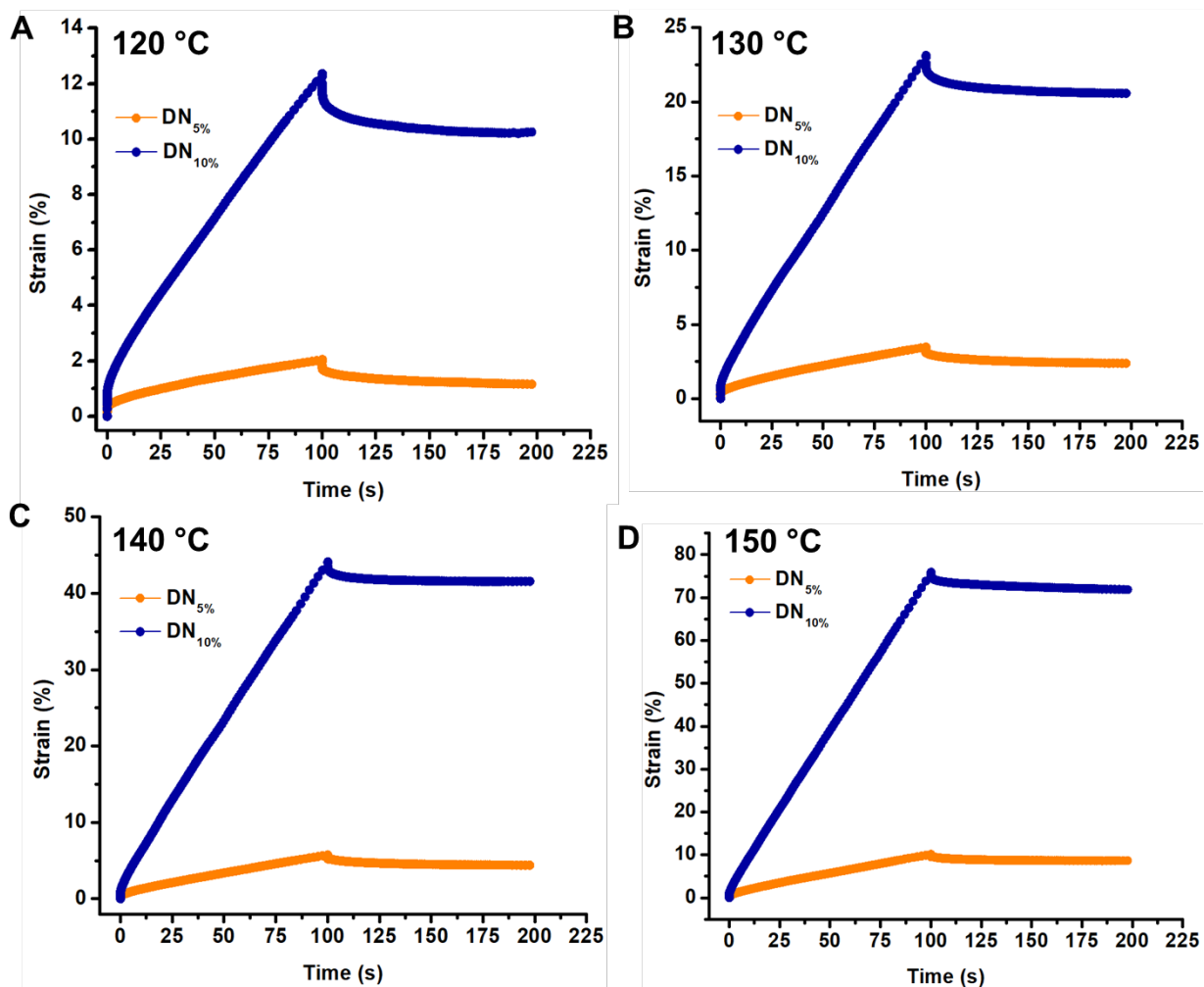
Creep-recovery experiments



**Figure S19.** Comparison of the creep-recovery experiments of SN<sub>5%</sub> and SN<sub>10%</sub> at A) 120 °C; B) 130 °C; C) 140 °C; D) 150 °C.

**Table S2.** Recovery strain% of the samples SN<sub>5%</sub> and SN<sub>10%</sub>

Sample - Temperature	Recovery strain
SN <sub>5%</sub> -120 °C	2.1 %
SN <sub>5%</sub> -130 °C	3.8 %
SN <sub>5%</sub> -140 °C	7.1 %
SN <sub>5%</sub> -150 °C	12.6 %
SN <sub>10%</sub> -120 °C	17.7 %
SN <sub>10%</sub> -130 °C	34.8 %
SN <sub>10%</sub> -140 °C	63.8 %
SN <sub>10%</sub> -150 °C	107.5 %

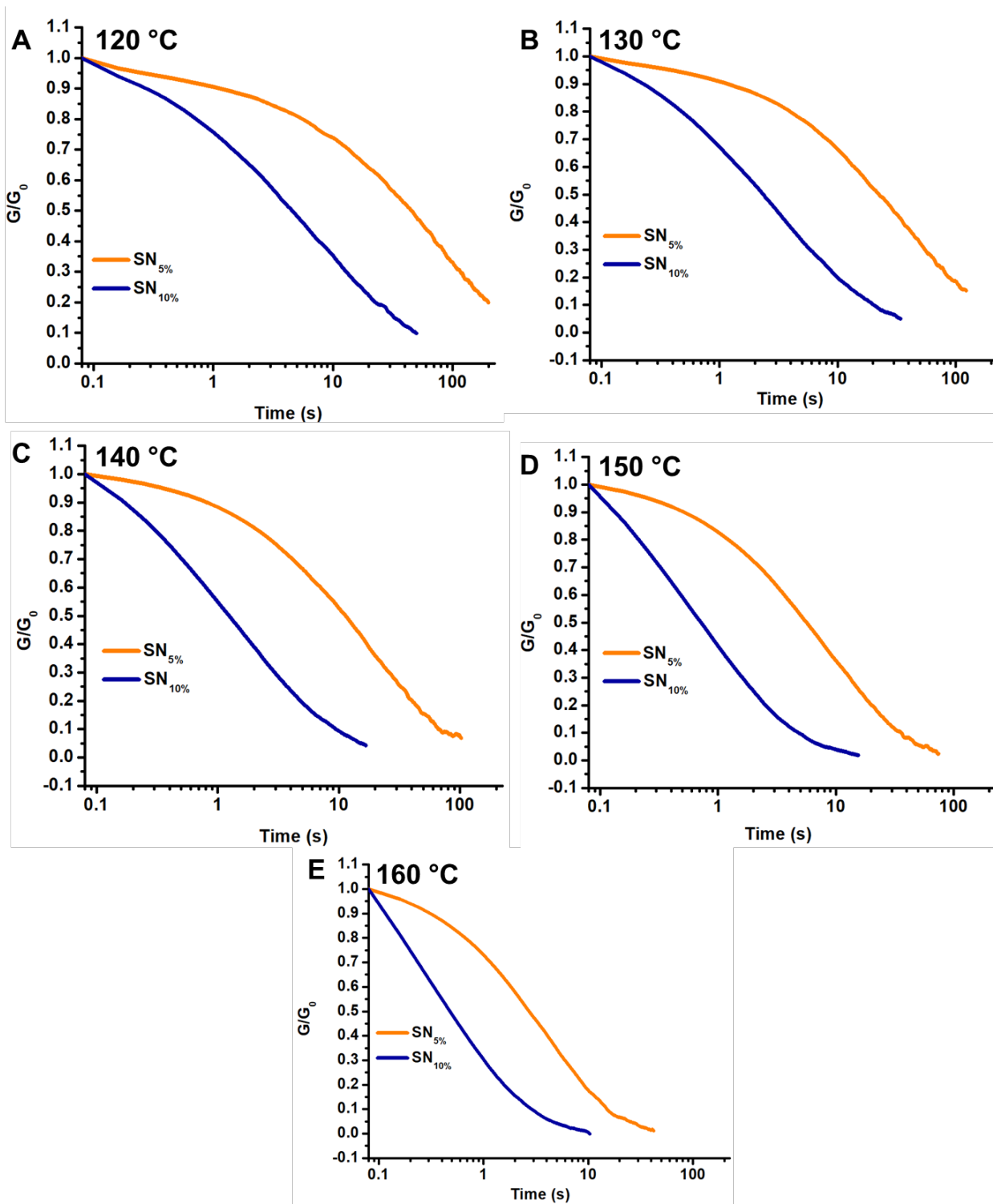


**Figure S20.** Comparison of the creep-recovery experiments of DN<sub>5%</sub> and DN<sub>10%</sub> at A) 120 °C; B) 130 °C; C) 140 °C; D) 150 °C.

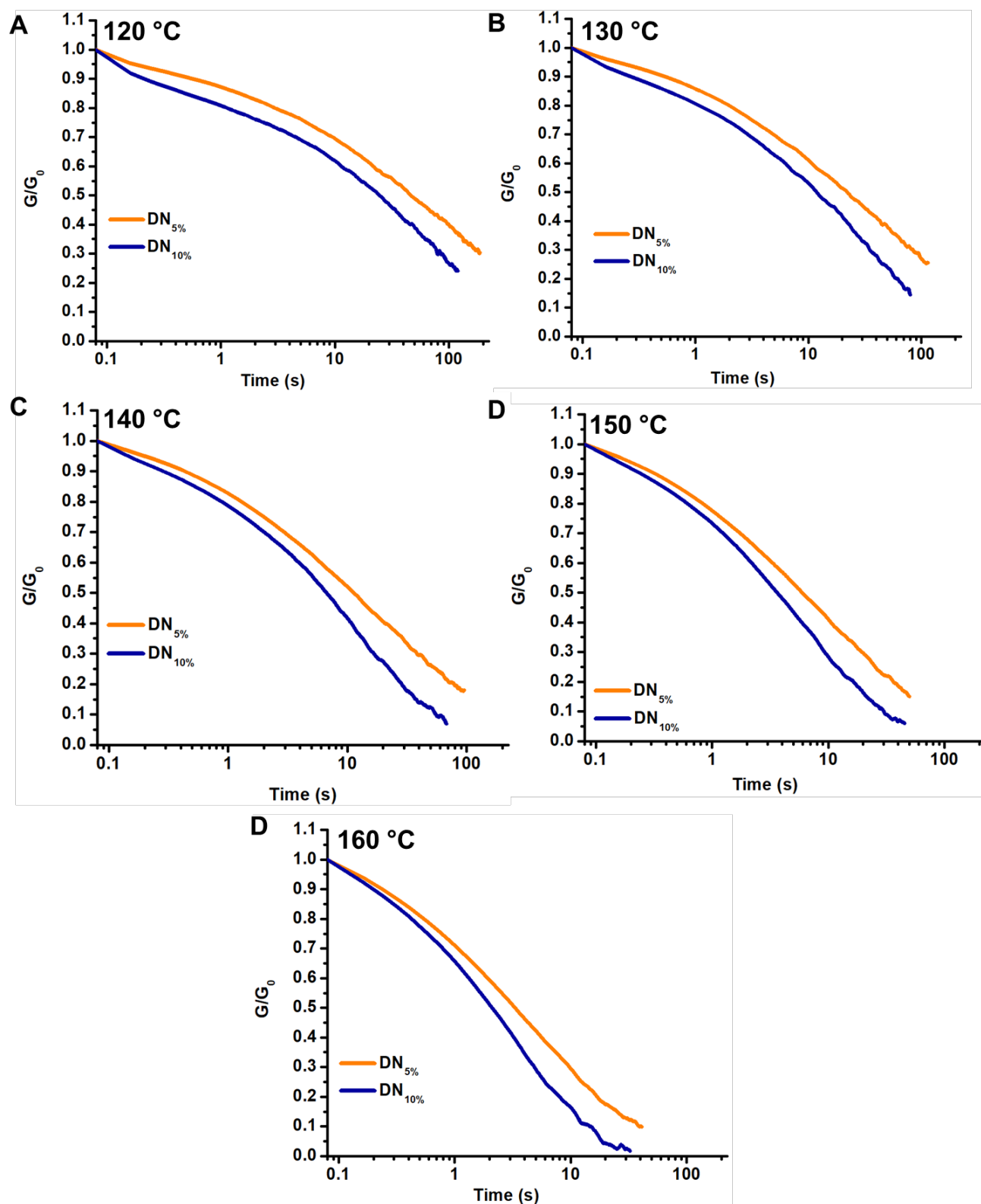
**Table S3.** Recovery strain% of the samples DN<sub>5%</sub> and DN<sub>10%</sub>

Sample - Temperature	Recovery strain
DN <sub>5%</sub> -120 °C	1.1 %
DN <sub>5%</sub> -130 °C	2.2 %
DN <sub>5%</sub> -140 °C	4.2 %
DN <sub>5%</sub> -150 °C	8.4 %
DN <sub>10%</sub> -120 °C	10.3 %
DN <sub>10%</sub> -130 °C	20.7 %
DN <sub>10%</sub> -140 °C	41.5 %
DN <sub>10%</sub> -150 °C	72.3 %

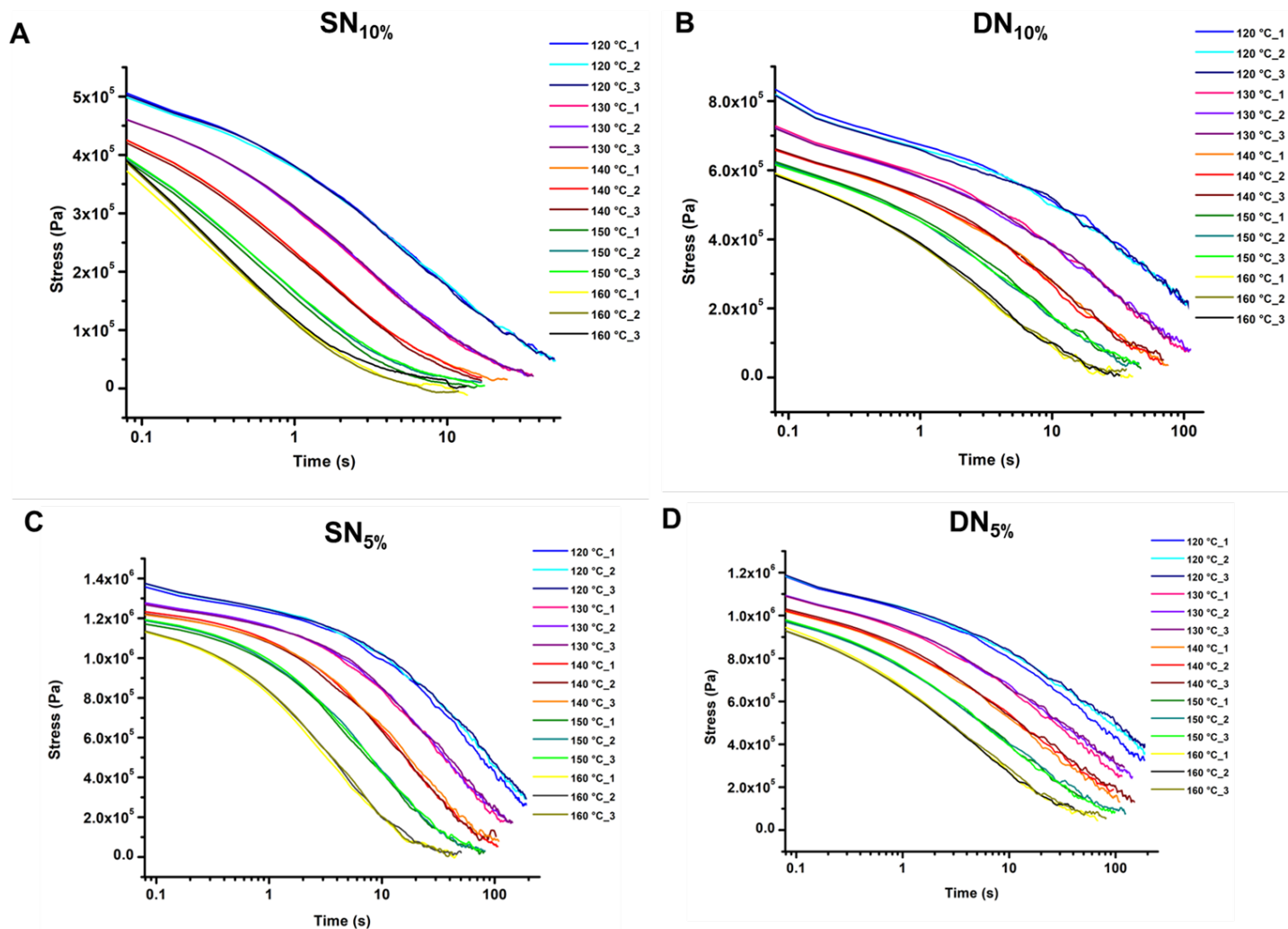
Stress relaxation



**Figure S21.** Comparison of the stress-relaxation experiments of SN<sub>5%</sub> and SN<sub>10%</sub> at A) 120 °C; B) 130 °C; C) 140 °C; D) 150 °C; E) 160 °C.



**Figure S22.** Comparison of the stress-relaxation experiments of DN<sub>5%</sub> and DN<sub>10%</sub> at A) 120 °C; B) 130 °C; C) 140 °C; D) 150 °C; E) 160 °C.



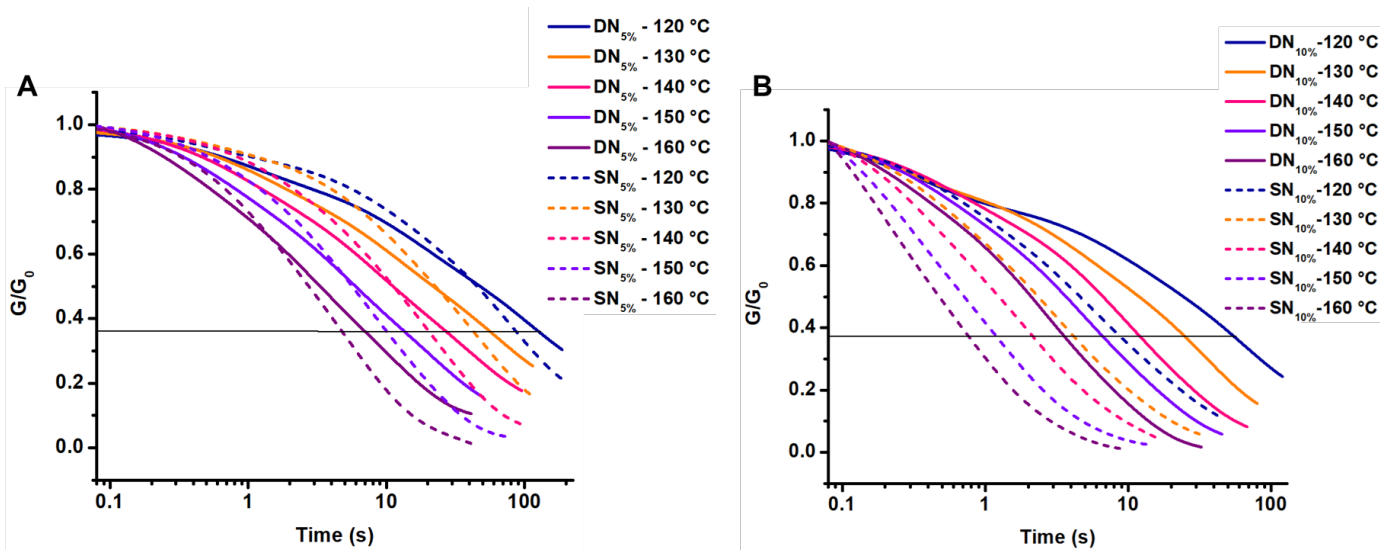
**Figure S23.** Triplicates of raw data for A)SN<sub>10%</sub>; B) DN<sub>10%</sub>; C)SN<sub>5%</sub>; D) DN<sub>5%</sub>.

Arrhenius relation (calculation of activation energy using a single Maxwell model)

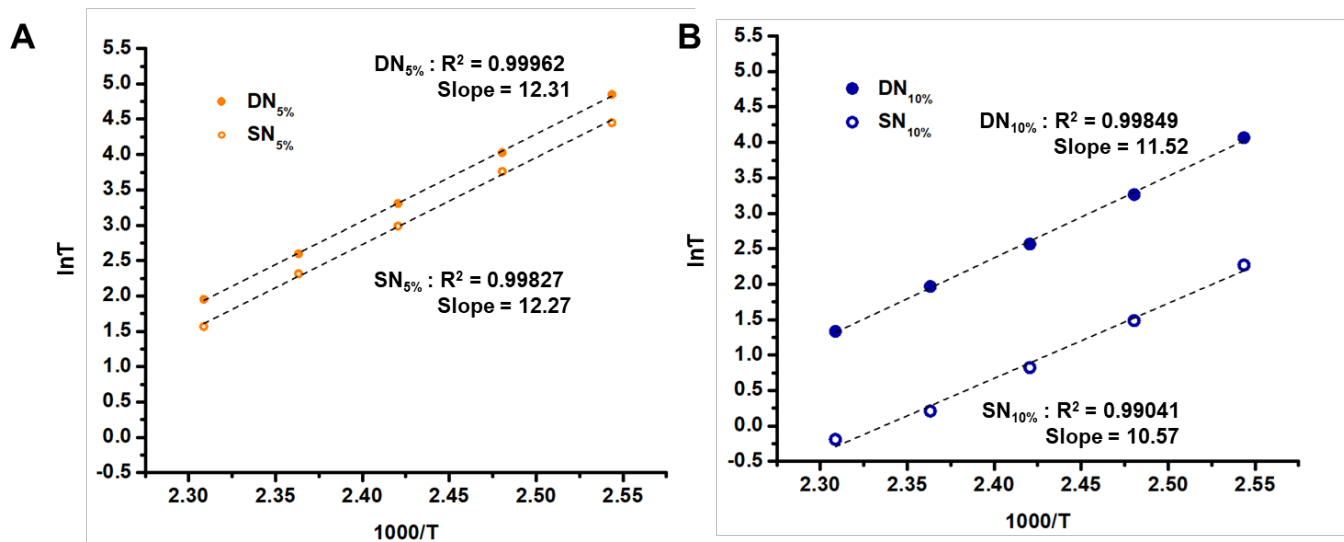
The characteristic relaxation time ( $\tau$ ) is the normalized stress relaxation at  $1/e$ , assuming a single Maxwell element as seen in literature examples.

$\tau = \text{time at } (1/e) = \text{time at } 0.37 \text{ normalized modulus}$

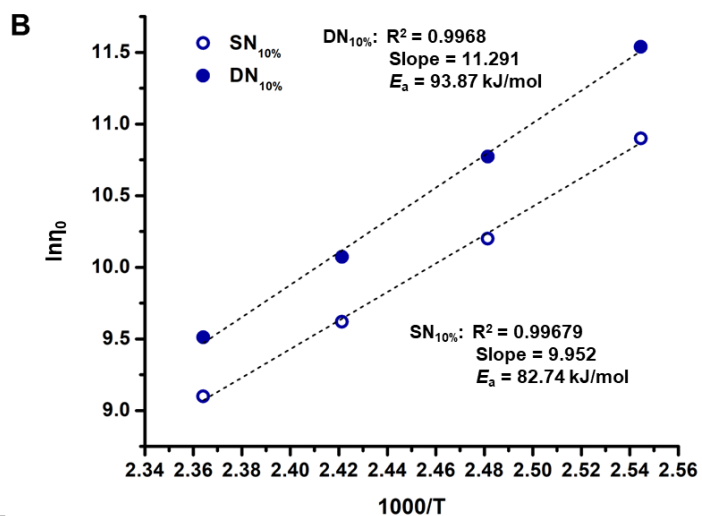
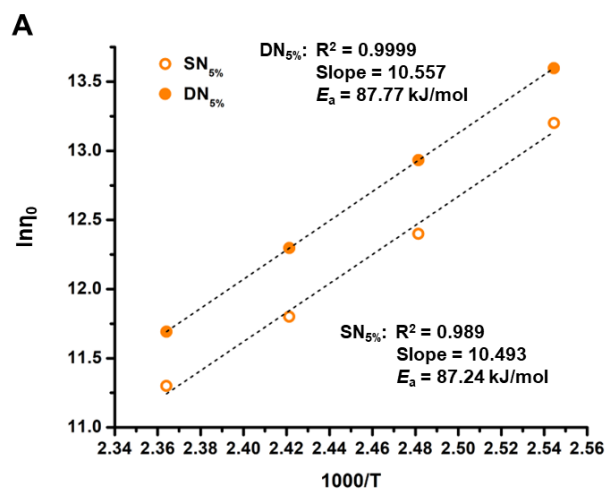
$$\ln(\tau) = E_a/RT + \ln(\tau_0) \dots\dots\dots (1)^{4,5}$$



**Figure S24.** Normalized stress relaxation curves fitted to three phase exponential decay for A) SN<sub>5%</sub> and DN<sub>5%</sub>; B) SN<sub>10%</sub> and DN<sub>10%</sub>.



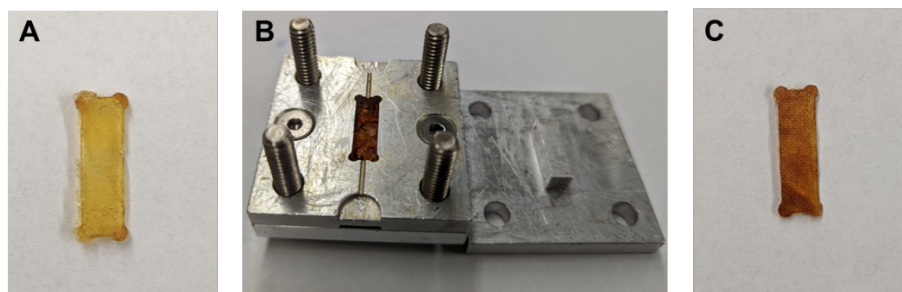
**Figure S25.** Calculation of activation energies of flow from stress relaxation A) DN<sub>5%</sub> and SN<sub>5%</sub>; B) DN<sub>10%</sub> and SN<sub>10%</sub>.



**Figure S26.** Activation energy of flow calculated from creep-recovery experiment following previous report A) DN<sub>5%</sub> and SN<sub>5%</sub>; B) DN<sub>10%</sub> and SN<sub>10%</sub>.<sup>6,7</sup>



## Reprocessing analysis



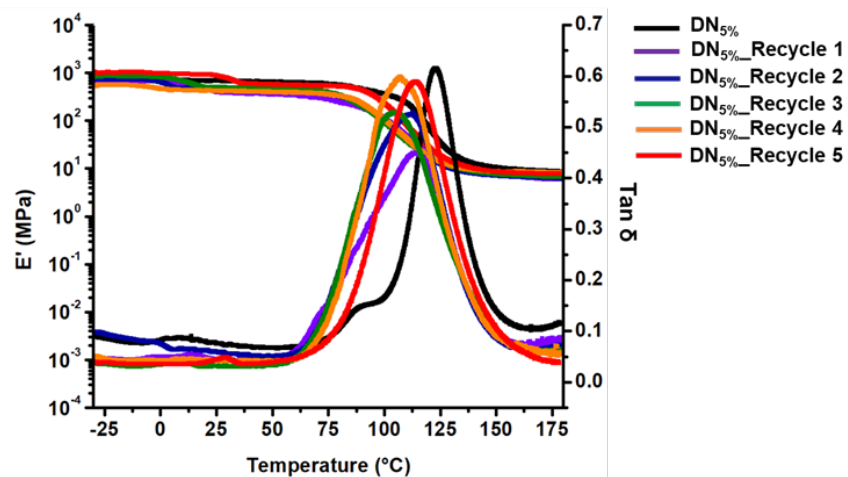
**Figure S27.** A) Sample before analysis; B) Shredded samples after analysis in a mold; C) Compression molded samples.

After analysis of the sample, the material was shredded into small pieces. The shredded sample was compression molded at 150 °C under an inert atmosphere for 2 h to yield the reprocessed sample.

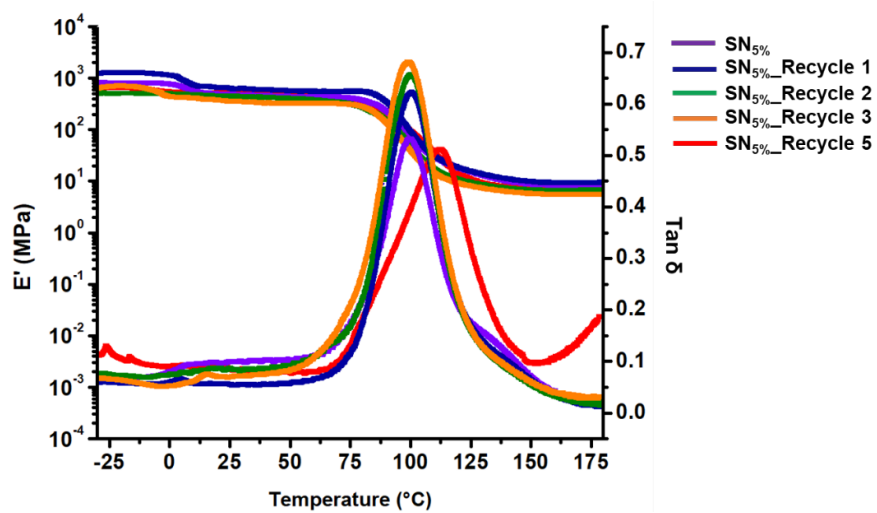
**Table S4.** Tabulated  $E'$  and  $T_g$  for different networks

Sample	$E'$ (175 °C)	$T_g$
SN <sub>10%</sub>	0.401 MPa	87 °C
DN <sub>10%</sub>	0.399 MPa	126 °C
SN <sub>5%</sub>	8.31 MPa	100 °C
SN <sub>5%</sub> _Recycle 1	9.13 MPa	113 °C
SN <sub>5%</sub> _Recycle 2	6.3 MPa	100 °C
SN <sub>5%</sub> _Recycle 3	5.14 MPa	99 °C
SN <sub>5%</sub> _Recycle 5	6.28 MPa	100 °C
DN <sub>5%</sub>	8.82 MPa	122 °C
DN <sub>5%</sub> _Recycle 1	6.68 MPa	114 °C
DN <sub>5%</sub> _Recycle 2	6.48 MPa	114 °C
DN <sub>5%</sub> _Recycle 3	6.75 MPa	104 °C
DN <sub>5%</sub> _Recycle 4	8.21 MPa	107 °C
DN <sub>5%</sub> _Recycle 5	7.91 MPa	114 °C

## DMA for reprocessing samples



**Figure S28.** Temperature ramp by DMA for DN<sub>5%</sub> over five recycling cycles.



**Figure S29.** Temperature ramp by DMA for SN<sub>5%</sub> over five recycling cycles.

## DSC thermograms

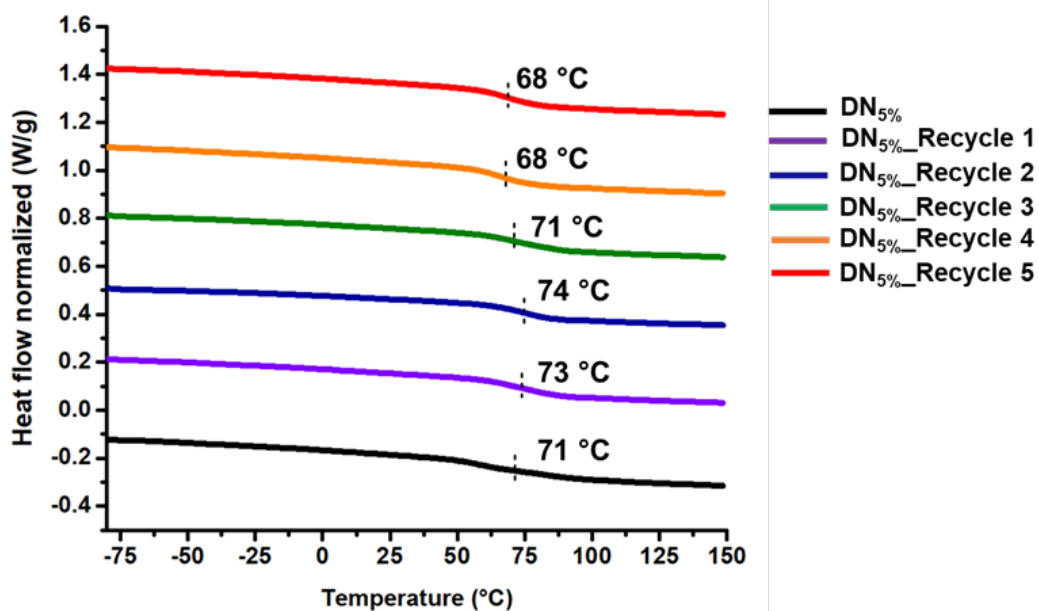


Figure S30. DSC thermograms of DN<sub>5%</sub> over five reprocessing cycles.

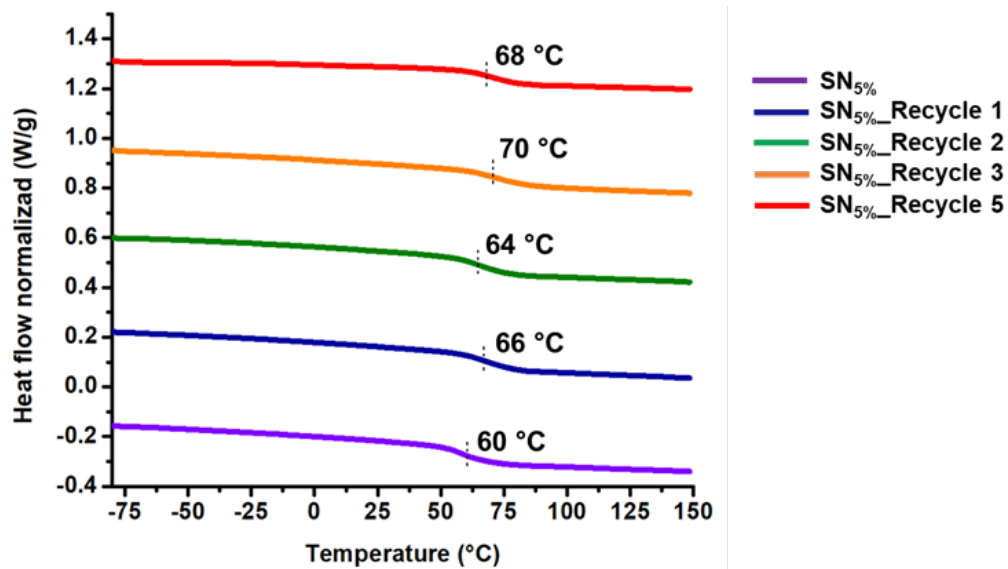


Figure S31. DSC thermograms of SN<sub>5%</sub> over five reprocessing cycles.

FT-IR spectroscopy

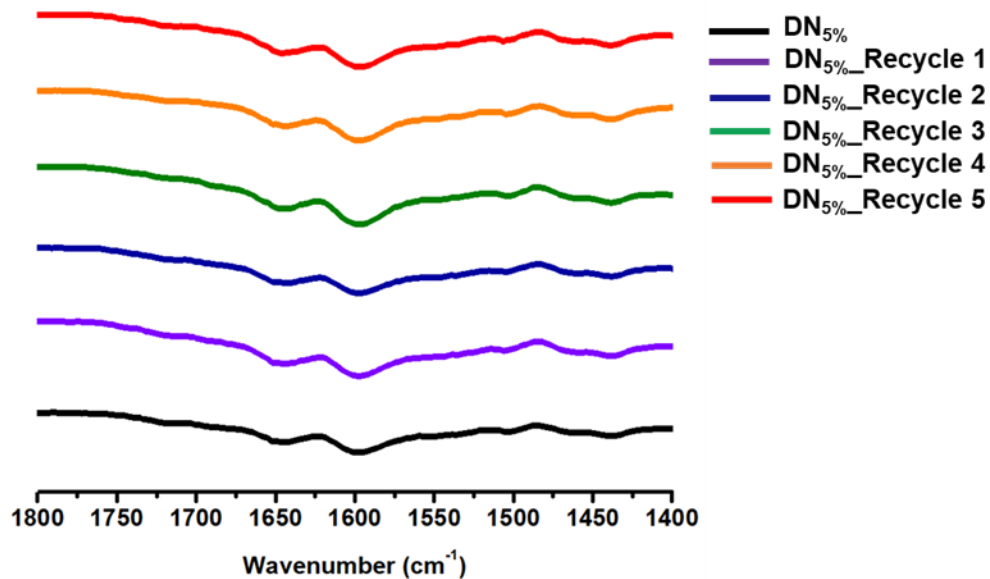


Figure S32. FT-IR spectra of DN<sub>5%</sub> over five reprocessing cycles.

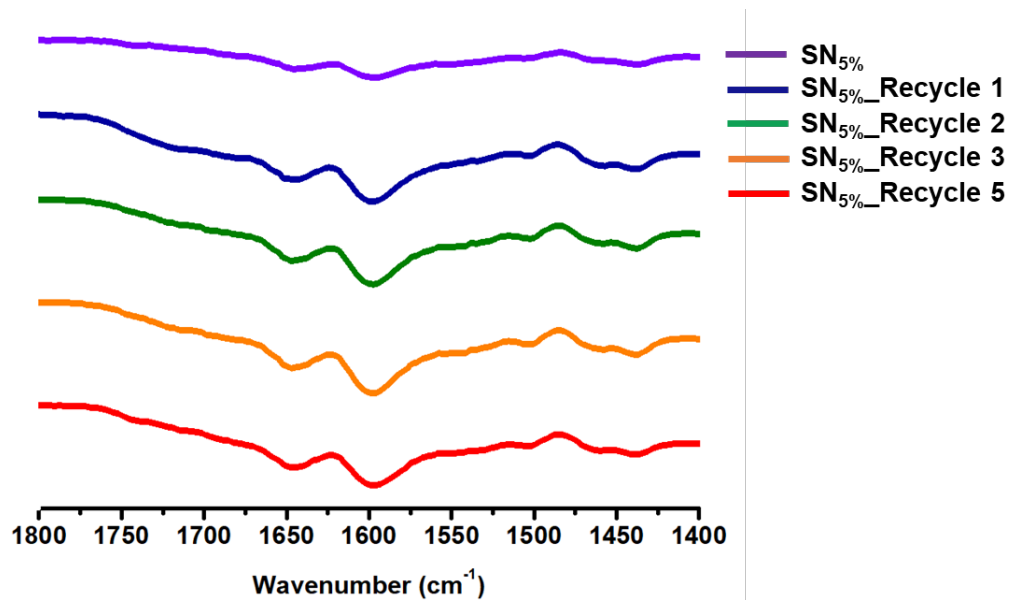
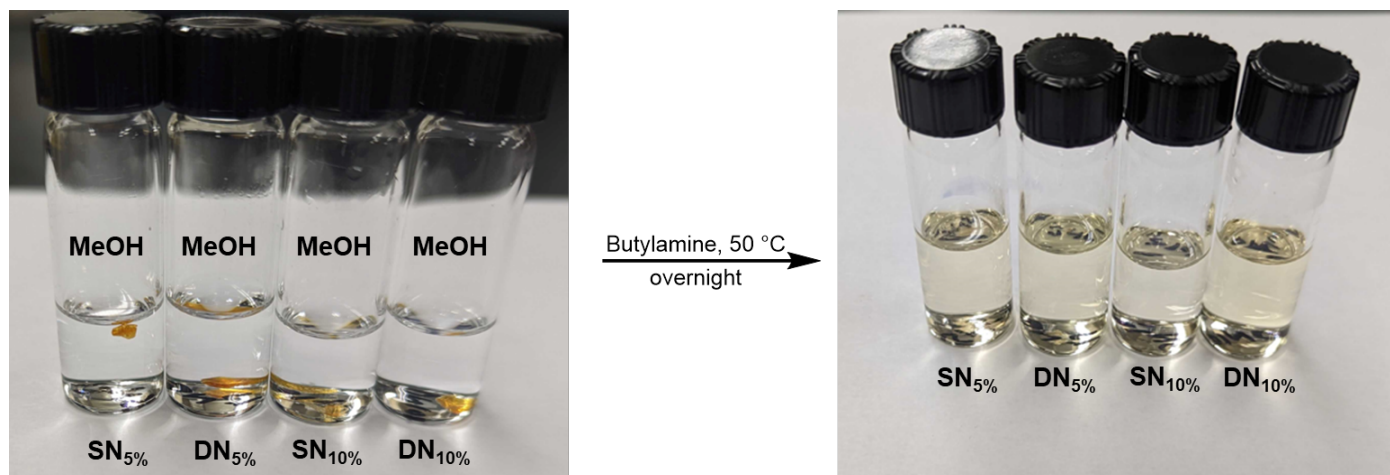


Figure S33. FT-IR spectra of SN<sub>5%</sub> over five reprocessing cycles.

## Chemical degradation studies

Butyl amine (1 mL) was added into the vial containing networks (20-30 mg) and MeOH (2 mL). The solution was heated at (50 °C) overnight to obtain a clear solution.



**Figure S34.** Images of chemical degradation studies.

## References

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