

## Supporting Information

# Mussel-like Surface Adhesion and Photoinduced Cooperative Deformation of Janus Particles

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**SI includes 15 pages, 15 figures, and 2 tables.**

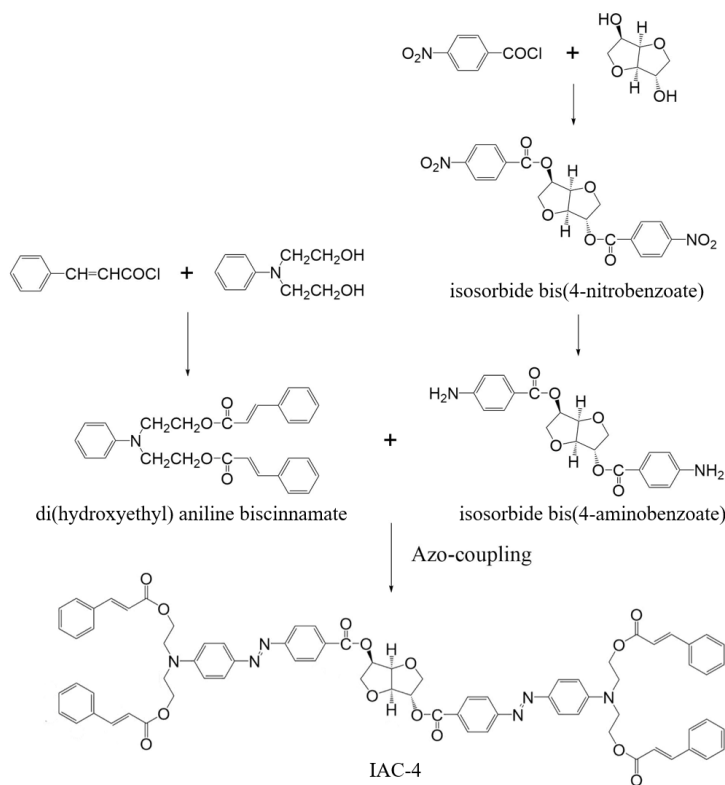
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## S1. Synthesis

**Materials.** Isosorbide, 4-nitrobenzoyl chloride, 2,6-pyridinedicarbonyl dichloride, and *bis*(3-aminopropyl)terminated poly(dimethylsiloxane) ( $\text{H}_2\text{N-PDMS-NH}_2$ ,  $M_n = 2500$ ) were purchased from Sigma-Aldrich. Cinnamoyl chloride and *N,N*-di(hydroxyethyl)-aniline were purchased from Alfa Aesar and used as received. Glacial acetic acid and concentrated sulfuric acid, analytical pure tetrahydrofuran (THF), dichloromethane (DCM), and *N,N*-dimethylformamide (DMF) were purchased commercially and used as received for the azo-coupling reaction and as the solvents. Poly(vinyl alcohol) (PVA) with the hydrolysis degree of 87.0%-89.0% (mol/mol) was purchased from Shanghai Aladdin biochemical technology. Ultrapure water (resistivity  $> 18.0 \text{ M}\Omega\cdot\text{cm}$ ) was supplied by a Milli-Q water purification system and used for all experiments. Other reagents were commercially available products and used without further purification.

The azo compound IAC-4 was synthesized through the route shown in Scheme S1 according to our previous paper,<sup>S1</sup> which is described below in detail.



**Scheme S1.** Synthesis route of IAC-4.

**Synthesis of isosorbide *bis*(4-nitrobenzoate).** Isosorbide (7.31 g, 0.05 mol) and triethylamine (15 mL) were dissolved in DMF (50 mL) in a reaction flask, and the mixture was stirred with ice-water bath cooling. 4-Nitrobenzoyl chloride was dissolved in DMF (100 mL) and added dropwise into the mixture. The reaction was carried out at the room temperature for 6 h. After the reaction was completed, the mixture was washed with saturated salt water and dried. White solid was obtained after washing with hot alcohol and distillation under vacuum.  $^1\text{H-NMR}$  ( $\text{CDCl}_3$ , 600 MHz,  $\delta$ , ppm): 8.35-8.12 (m, 8H, -ArH), 5.43-5.41 (q, 1H, -COO-CH-, isosorbide), 5.37-5.36 (d, 1H, -COO-CH-, isosorbide), 5.01-4.99 (t, 1H, -CH<sub>2</sub>-O-CH, isosorbide), 4.65-4.64 (d, 1H, -CH<sub>2</sub>-O-CH, isosorbide), 4.04-4.02 (q, 2H, -CH<sub>2</sub>-O-CH, isosorbide), 3.97-3.91 (m, 2H, -CH<sub>2</sub>-O-CH, isosorbide).

**Synthesis of isosorbide *bis*(4-aminobenzoate).** Isosorbide *bis*(4-nitrobenzoate) (18.8 g, 0.042 mol), sodium sulfide nonahydrate (57.6 g, 0.24 mol) and ammonium chloride (18.0 g, 0.34 mol) were dissolved in water (100 mL) and alcohol (200 mL). The mixture was heated to the reflux temperature (about 75 °C) and reacted for 6 h. After the reaction, the mixture was poured into plenty of water and the precipitate was collected by filtration. The product (isosorbide *bis*(4-aminobenzoate)) was dried in a vacuum oven at 70 °C for 24 h. IR (KBr,  $\text{cm}^{-1}$ ): 3442, 3353, 3239 (m, N-H), 1689 (m, C=O), 1597, 1516 (m, benzene ring), 1278 (m, C-N), 1117 (m, C-O-C).  $^1\text{H-NMR}$  (DMSO, 600 MHz,  $\delta$ , ppm): 7.62-7.58 (q, 4H, -ArH, meso to NH<sub>2</sub>), 6.54-6.51 (t, 4H, -ArH, *ortho* to NH<sub>2</sub>), 6.01-5.99 (br, 4H, -NH<sub>2</sub>), 5.22-5.21 (q, 1H, -COO-CH-, isosorbide), 5.18 (d, 1H, -COO-CH-, isosorbide), 4.86-4.84 (t, 1H, -CH<sub>2</sub>-O-CH, isosorbide), 4.50 (d, 1H, -CH<sub>2</sub>-O-CH, isosorbide), 3.91-3.82 (s, 4H, -CH<sub>2</sub>-O-CH, isosorbide).

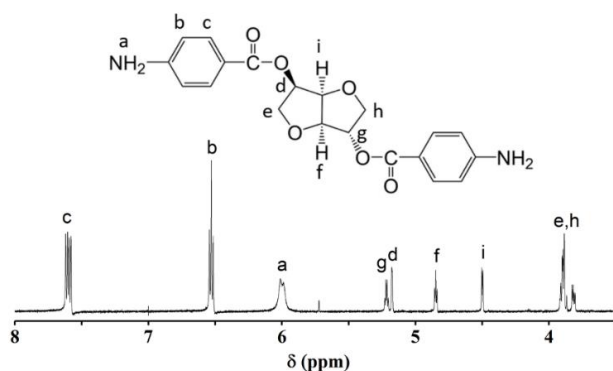
**Synthesis of *di*(hydroxyethyl)aniline *biscinnamate*.** *N,N*-*di*(hydroxyethyl)aniline (18.1 g, 0.1 mol) and triethylamine (42 mL) were dissolved in DCM (100 mL) and the mixture was stirred in a reaction flask cooling with an ice-water bath. Cinnamoyl chloride (40 g, 0.24 mol) was added dropwise into the mixture. The mixture was reacted under the ice-water bath cooling for 24 h. After the reaction, the mixture was washed with saturated salt water and then the organic solvent phase was dried with anhydrous  $\text{MgSO}_4$ . Yellow thick liquid was obtained by column chromatography (eluent: DCM/EA=10/1). IR (KBr,

cm<sup>-1</sup>): 3060 (m, =CH-), 3000-2800 (m, -CH<sub>2</sub>, -CH), 1712 (m, C=O), 1636 (m, -CH=CH-), 1599, 1577, 1505, 1450 (m, benzene ring). <sup>1</sup>H-NMR (CDCl<sub>3</sub>, 600 MHz, δ, ppm): 7.59-7.57 (d, 2H, -CH=CH-Ph), 7.52-7.51 (q, 4H, ArH, *ortho* to -CH=CH-), 7.39-7.37 (s, 6H, ArH, *meso* and *para* to -CH=CH-), 7.29-7.25 (q, 2H, ArH, *meso* to -N(CH<sub>2</sub>)<sub>2</sub>), 6.87-6.86 (d, 2H, ArH, *ortho* to -N(CH<sub>2</sub>)<sub>2</sub>), 6.77-6.76 (t, 1H, ArH, *para* to -N(CH<sub>2</sub>)<sub>2</sub>), 6.46-6.44 (d, 2H, -CH=CH-Ph), 4.43-4.41 (t, 4H, -O-CH<sub>2</sub>-), 3.77-3.75 (t, 4H, -N-CH<sub>2</sub>).

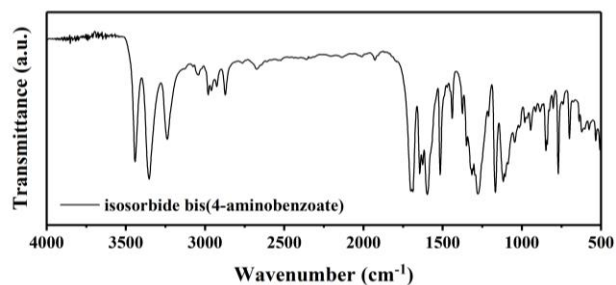
**Synthesis of IAC-4.** IAC-4 was synthesized by azo-coupling reaction between isosorbide *bis*(4-aminobenzoate) and *N,N*-di(hydroxyethyl)aniline *biscinnamate*. *N,N*-di(hydroxyethyl)aniline *biscinnamate* (2.05 g, 0.0047 mol) was dissolved in DMF (100 mL) and stirred in a reaction flask cooling with ice-water bath. Isosorbide *bis*(4-aminobenzoate) (0.59 g, 0.0016 mol) was mixed with glacial acetic acid (9 mL) and sulfuric acid (0.9 mL) in an ice-water bath. The mixture was stirred at 0 °C for 2 h until no solid existed. The diazonium salt was prepared by adding an aqueous solution of NaNO<sub>2</sub> (0.31 g, 0.0045 mol in 0.9 mL water) into the mixture of isosorbide *bis*(4-aminobenzoate). Homogeneous solution obtained by stirring and ice-water bath cooling was added dropwise into the DMF solution of *N,N*-di(hydroxyethyl)aniline *biscinnamate*. The solution was reacted at 0 °C for 12 h. After the reaction, the mixture was added into plenty of water, and the precipitate was collected by filtration. Red solid was obtained through column chromatography (eluent: DCM/EA=10/1). IR (KBr, cm<sup>-1</sup>): 3060 (m, =CH-), 3000-2800 (m, -CH<sub>2</sub>, -CH), 1712 (m, C=O), 1635 (m, -CH=CH-), 1597, 1512, 1449 (m, benzene ring), 1266 (m, C-N). <sup>1</sup>H-NMR (CDCl<sub>3</sub>, 600 MHz, δ, ppm): 8.13-8.20 (d × d, 2H, ArH, *ortho* to -COO-), 7.98-7.95 (q, 4H, ArH, *meso* to -COO-), 7.91-7.87 (q, 4H, ArH, *meso* to -N(CH<sub>2</sub>)<sub>2</sub>), 7.68-7.65 (d, 4H, -CH=CH-Ph), 7.50-7.49 (d, 8H, ArH, *ortho* to -CH=CH-), 7.39-7.34 (s, 12H, ArH, *meso* and *para* to -CH=CH-), 6.97-6.95 (q, 4H, ArH, *ortho* to N(CH<sub>2</sub>)<sub>2</sub>), 6.44-6.41 (q, 4H, -CH=CH-Ph), 5.54-5.53 (d, 1H, -COO-CH-, isosorbide), 5.48-5.45 (q, 1H, -COO-CH-, isosorbide), 5.12-5.11 (t, 1H, -CH<sub>2</sub>-O-CH-, isosorbide), 4.74 (d, 1H, -CH<sub>2</sub>-O-CH-, isosorbide), 4.49-4.47 (t, 8H, -CO-CH<sub>2</sub>CH<sub>2</sub>-N-), 4.19-4.08 (s, 4H, -CH<sub>2</sub>-O-CH-, isosorbide), 3.88-3.86 (t, 8H, -CO-CH<sub>2</sub>CH<sub>2</sub>-N-). DSC: *T*<sub>g</sub> = 57 °C. The molar absorption coefficient of IAC-4 is 3.09×10<sup>4</sup> L mol<sup>-1</sup> cm<sup>-1</sup>.

**Synthesis of H<sub>2</sub>pdca-PDMS.** The polydimethylsiloxane oligomer (H<sub>2</sub>pdca-PDMS) was synthesized according to the literature.<sup>S2</sup> Triethylamine (Et<sub>3</sub>N, 2 mL) was added into a solution of H<sub>2</sub>N-PDMS-NH<sub>2</sub> (12.5 g, M<sub>n</sub> = 2500) in anhydrous DCM (40 mL) cooling with an ice bath under argon atmosphere. After stirring for 2 h, a solution of 2,6-pyridinedicarbonyl dichloride (1.02 g, 5 mmol) in DCM (10 mL) was added dropwise into the reaction flask. After continually stirring for 2 h with the ice bath cooling, the mixture was heated to 50 °C and stirred for 2 days. After the reaction, the solution was concentrated to 1/3 of its original volume and then 60 mL MeOH was added into it. White viscous liquid precipitated and the mixture was set aside for 30 min. After decanting the upper clear solution, 20 mL DCM was added to dissolve the product. The dissolution-precipitation-decantation process was repeated for three times and the final product was obtained through vacuum evaporation. <sup>1</sup>H-NMR (CDCl<sub>3</sub>, 600 MHz, δ, ppm): 8.34-8.35 (d, 2H, ArH, *metho* to N), 7.99-8.02 (t, 1H, ArH, *para* to N), 7.71 (br, 2H, -NH-), 3.47-3.50 (m, 4H, -CH<sub>2</sub>-, α to -NH-), 1.67-1.68 (m, 4H, -CH<sub>2</sub>-, β to -NH-), 0.61-0.63 (m, 4H, -CH<sub>2</sub>-, γ to -NH-). GPC (THF): M<sub>w</sub> = 29700 g mol<sup>-1</sup>, M<sub>n</sub> = 17100 g mol<sup>-1</sup>, M<sub>w</sub>/M<sub>n</sub> = 1.7.

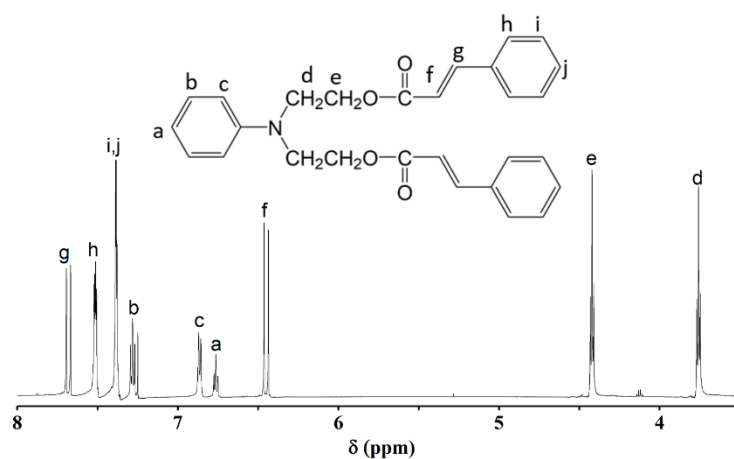
## S2. Charaterization



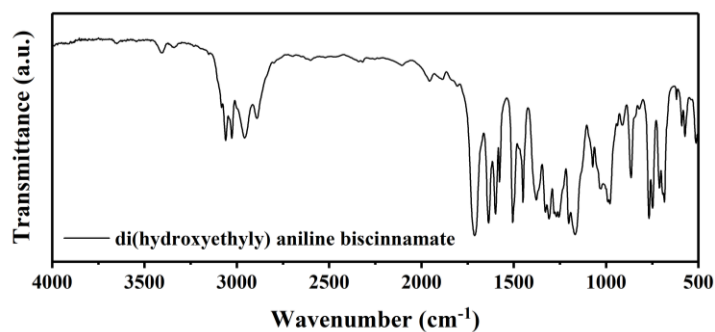
**Fig. S1.** <sup>1</sup>H-NMR spectrum of isosorbide *bis*(4-aminobenzoate).



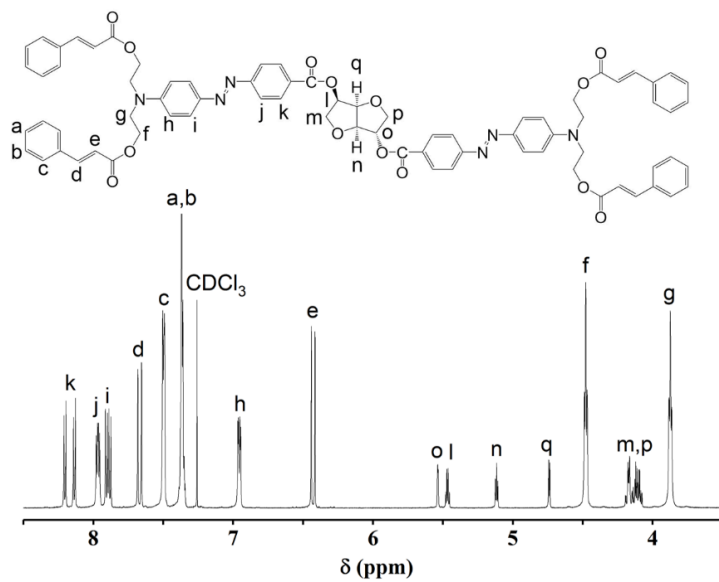
**Fig. S2.** FT-IR spectrum of isosorbide *bis*(4-aminobenzoate).



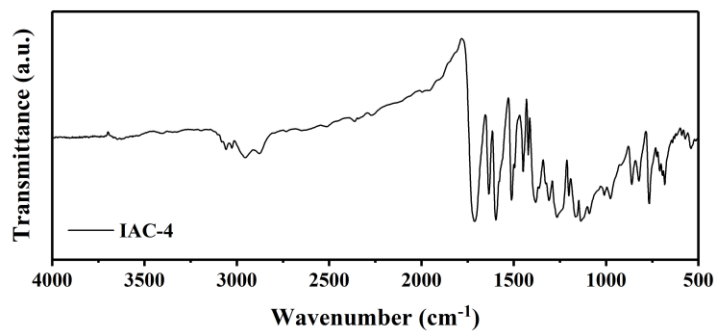
**Fig. S3.**  $^1\text{H}$ -NMR spectrum of *N,N*-di(hydroxyethyl)aniline *biscinnamate*.



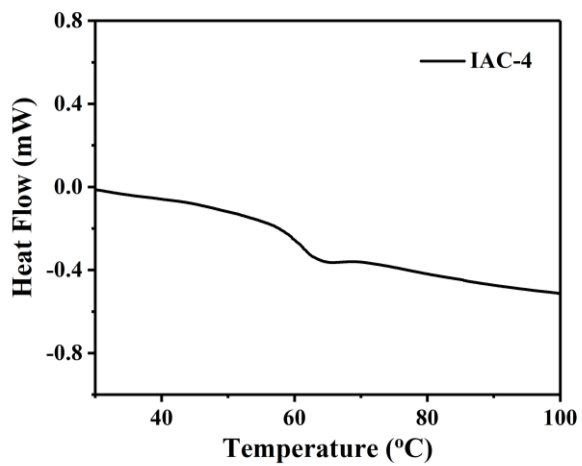
**Fig. S4.** FT-IR spectrum of *N,N*-di(hydroxyethyl)aniline *biscinnamate*.



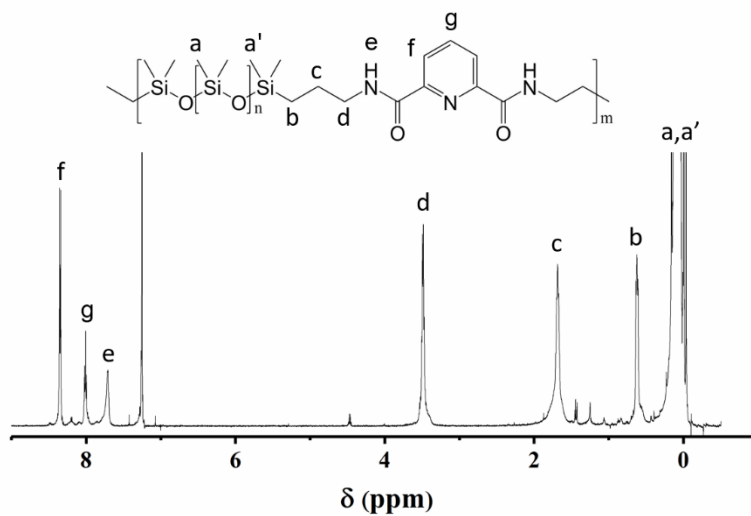
**Fig. S5.**  $^1\text{H}$ -NMR spectrum of IAC-4.



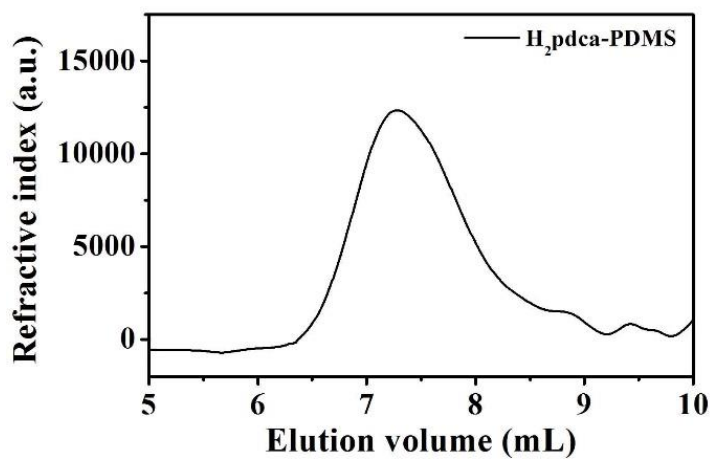
**Fig. S6.** FT-IR spectrum of IAC-4.



**Fig. S7.** DSC curve of IAC-4,  $T_g = 57^{\circ}\text{C}$ .

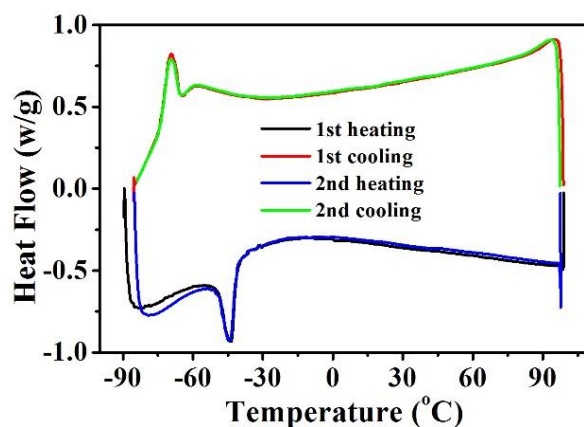


**Fig. S8.**  $^1\text{H}$ -NMR spectrum of  $\text{H}_2\text{pdca-PDMS}$ .

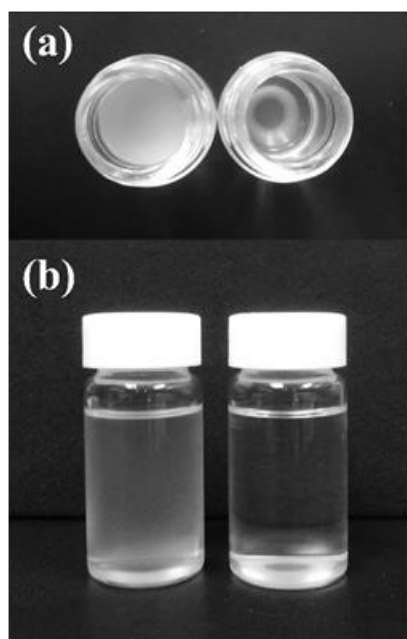


**Fig. S9.** GPC curve of  $\text{H}_2\text{pdca-PDMS}$  with THF as the eluent,  $M_w = 29700 \text{ g mol}^{-1}$ ,  $M_n = 17100 \text{ g mol}^{-1}$ ,  $M_w/M_n = 1.7$ .

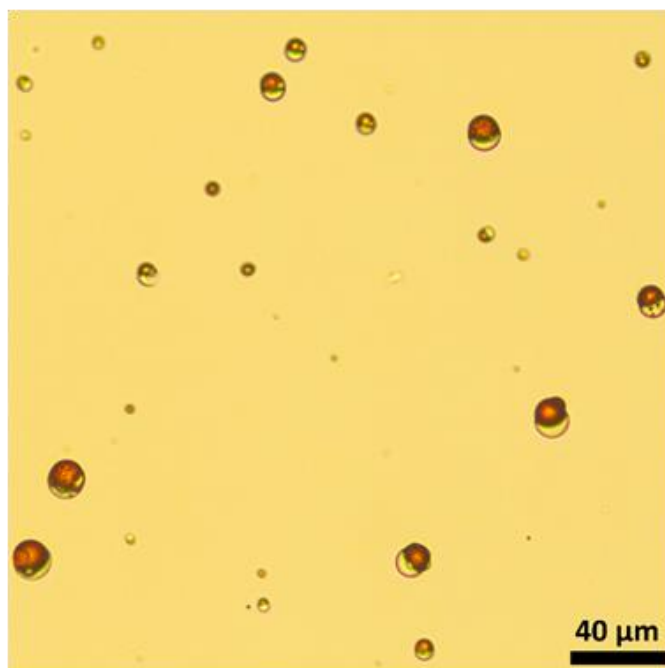




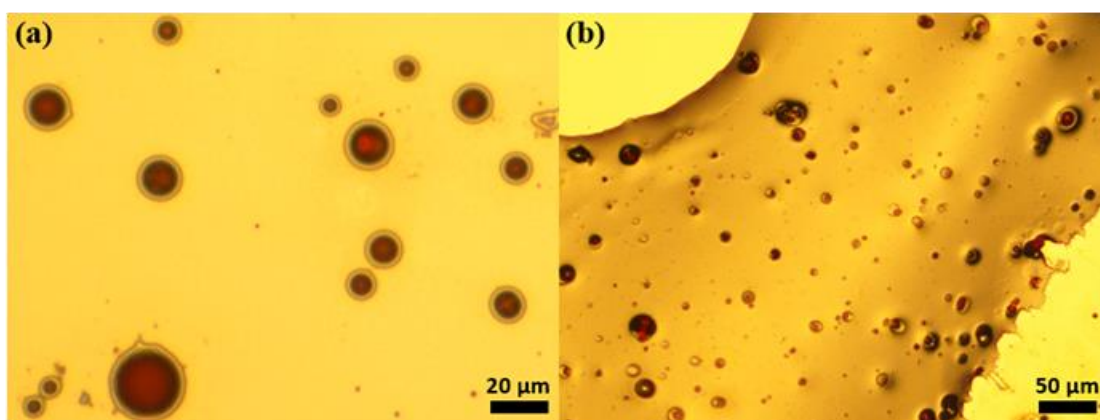
**Fig. S10.** DSC curve of H<sub>2</sub>pdca-PDMS. The exothermal peak at  $-64.7\text{ }^{\circ}\text{C}$  and endothermic peak at  $-43.7\text{ }^{\circ}\text{C}$  are consistent with the reported values.<sup>S2</sup>



**Fig. S11.** (a) Top-view and (b) side-view photographs of the suspensions of H<sub>2</sub>pdca-PDMS particles prepared through the evaporation of DCM from H<sub>2</sub>pdca-PDMS/DCM droplets dispersed in a PVA aqueous solution (2.5 wt%), prepared with stirring at  $35\text{ }^{\circ}\text{C}$  (left) and without stirring at  $5\text{ }^{\circ}\text{C}$  (right). The sedimentation can be clearly seen in the images of the suspension obtained under conditions without stirring at  $5\text{ }^{\circ}\text{C}$  (right).



**Fig. S12.** OM images of IAC-4/H<sub>2</sub>pdca-PDMS JPs prepared with stirring in an ice-bath.



**Fig. S13** Typical OM images, (a) JPs after washing off PVA, (b) morphology when PVA was not completely washed off. As shown in (a), the substrate is clean with a few spots or patches. In contrast, (b) shows that when PVA was not completely washed off, a thin PVA film formed on the substrate after drying and the JPs were embedded in it. The morphology and photoinduced deformation of the JPs cannot be characterized for a sample with residual PVA.

### S3. Calculation of the Phase Diagram

According to Flory-Huggins theory, the dimensionless mixing free energy per lattice site volume for the ternary system reads:<sup>S3</sup>

$$\frac{f}{kT} = \frac{\phi_1}{N_1} \ln \phi_1 + \frac{\phi_2}{N_2} \ln \phi_2 + \frac{\phi_3}{N_3} \ln \phi_3 + \chi_{12} \phi_1 \phi_2 + \chi_{13} \phi_1 \phi_3 + \chi_{23} \phi_2 \phi_3 \quad (S1)$$

Here,  $\phi_i$  and  $N_i$  represent volume fraction and relative degree of polymerization or size, with suffixes 1, 2, and 3 indicating the azo molecular glass (IAC-4), the PDMS oligomer (H<sub>2</sub>pdca-PDMS), and the solvent (DCM).  $\chi_{ij}$  ( $i = 1, 2, 3; j = 1, 2, 3$ ) are the monomeric binary interaction parameters and  $kT$  is the thermal energy. The relative degree of polymerization of the PDMS oligomer (H<sub>2</sub>pdca-PDMS) was obtained using the number-average molecular weight measured by GPC and volume per unit calculated by summing up the volumes of groups in the unit. For the azo molecular glass (IAC-4), the relative size was directly calculated by summing up the volumes of groups contained in the molecule. The volumes of the groups and the solvents were taken from literature.<sup>S4</sup> By normalizing the solvent molecular volume,  $N_1 = 14.2$ ,  $N_2 = 210.5$ ,  $N_3 = 1$  were obtained.  $\chi$ -values were estimated using the relation,<sup>S5</sup>

$$\chi_{12} = \alpha \frac{v_1}{RT} \left( (\delta_{1,d} - \delta_{2,d})^2 + 0.25(\delta_{1,p} - \delta_{2,p})^2 + 0.25(\delta_{1,hb} - \delta_{2,hb})^2 \right) \quad (S2)$$

where  $\delta_{i,d}$ ,  $\delta_{i,p}$ , and  $\delta_{i,hb}$  are the dispersive, polar and hydrogen-bonding Hansen parameters of the substance  $i$ ,  $v_1$  is the solvent molar volume and  $R$  is the gas constant. Previous study has suggested that by using  $\alpha = 0.6$  in equation S2 for minimum average absolute deviations, the procedure is proved to perform well for solutions containing polar and hydrogen-bonding compounds.<sup>S6</sup> The Hansen parameters of DCM were obtained from the literature.<sup>S4</sup> By the method of Hoftyzer and van Krevelen,<sup>S7</sup> the Hansen parameters of IAC-4 and H<sub>2</sub>pdca-PDMS were estimated by

$$\begin{aligned} \delta_d &= \frac{\sum F_{di}}{v} \\ \delta_p &= \frac{\sqrt{\sum F_{pi}^2}}{v} \end{aligned} \quad (S3)$$

$$\delta_h = \frac{\sqrt{\sum E_{hi}}}{V}$$

where  $F_{di}$ ,  $F_{pi}$ , and  $E_{hi}$  are the dispersive force, polar force, and hydrogen bonding energy of group  $i$ , which were obtained from literature.<sup>S7</sup> Thus, all  $\chi$ -values were estimated and all input parameters for Equation S1 were summarized in Table S1.

**Table S1.** Input data of the IAC-4/H<sub>2</sub>pdca-PDMS/DCM ternary system for calculation with the Flory-Huggins model.

	<b>IAC-4</b>	<b>H<sub>2</sub>pdca-PDMS</b>	<b>DCM</b>	<b>N</b>
<b>IAC-4</b>	$\chi_{11} = 0$	$\chi_{12} = 0.80$	$\chi_{13} = 0.19$	14.2
<b>H<sub>2</sub>pdca-PDMS</b>	$\chi_{21} = 0.80$	$\chi_{22} = 0$	$\chi_{23} = 0.31$	210.5
<b>DCM</b>	$\chi_{31} = 0.19$	$\chi_{32} = 0.31$	$\chi_{33} = 0$	1

**Table S2.** Critical values of the binary interaction parameters for the IAC-4/H<sub>2</sub>pdca-PDMS /DCM ternary system.

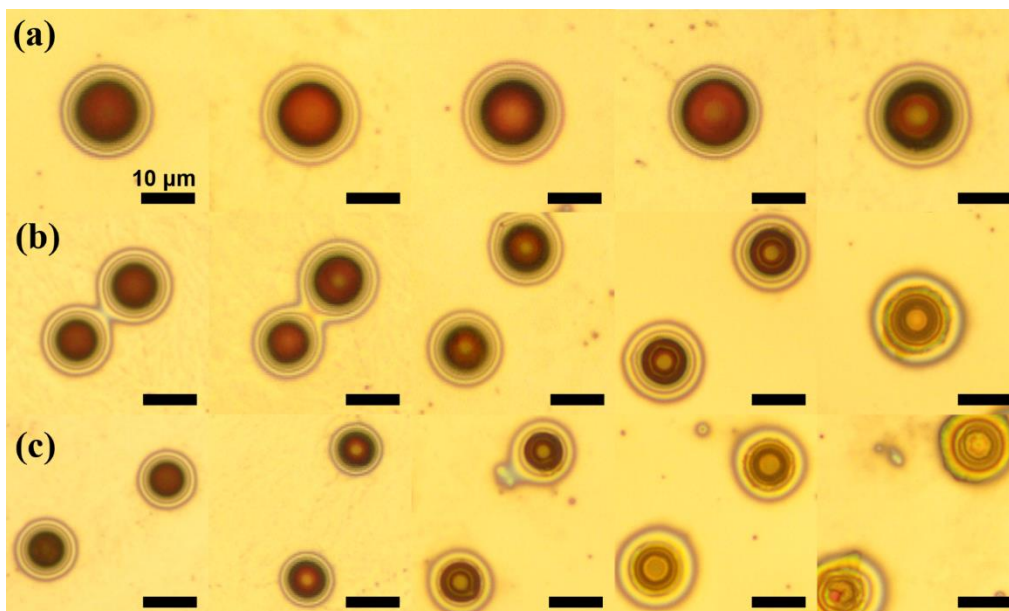
	<b>IAC-4</b>	<b>H<sub>2</sub>pdca-PDMS</b>	<b>DCM</b>
<b>IAC-4</b>	–	$\chi_{12,c} = 0.06$	$\chi_{13,c} = 0.80$
<b>H<sub>2</sub>pdca-PDMS</b>	$\chi_{21,c} = 0.06$	–	$\chi_{23,c} = 0.57$
<b>DCM</b>	$\chi_{31,c} = 0.80$	$\chi_{32,c} = 0.57$	–

In order to identify the driving force for the demixing of this ternary blend, the critical values of all binary interaction parameters were calculated as  $\chi_{ij,c} = \frac{1}{2} \left( \frac{1}{\sqrt{N_i}} + \frac{1}{\sqrt{N_j}} \right)^2$ , which were listed in Table S2. Comparing the interaction parameters in Table S1 with the corresponding values in Table S2 reveals that the repulsion between the azo molecular glass (IAC-4) and the PDMS oligomer (H<sub>2</sub>pdca-PDMS) is the driving force for liquid-liquid phase separation, since  $\chi_{12} \gg \chi_{12,c}$ . In contrast, the fact that  $\chi_{13} < \chi_{13,c}$  and  $\chi_{23} < \chi_{23,c}$  demonstrates that the amorphous materials show the reasonable to good affinity with the solvent. The positive tilt of the tie lines in the ternary phase diagram (Figure 4a,

in the main text) reflects a preference for the solvent to reside in IAC-4-rich phase in the ternary blends.

The ternary phase diagram given in Figure 4 in main text was calculated as follows. The spinodal curve (blue) was obtained by solving  $(\partial_{\phi_1\phi_1}f)(\partial_{\phi_2\phi_2}f) - (\partial_{\phi_1\phi_2}f)^2 = 0$ . The binodal line (red) and the tie-lines (violet) were obtained numerically using the common tangent construction.

#### S4. Deformation of JPs Induced by Circularly Polarized Light

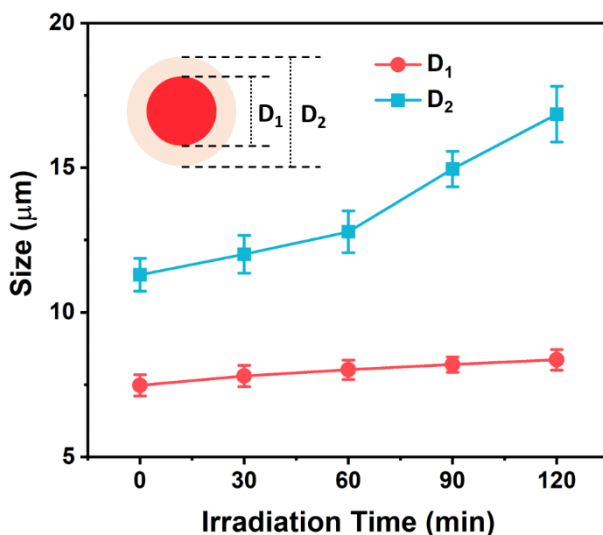


**Figure S14.** The reflection-mode OM images of the IAC-4/H<sub>2</sub>pdca-PDMS (w/w = 1/1) Janus particles with the IAC-4 part on the scale of (a) 11 μm, (b) 7.5 μm, and (c) 6 μm after irradiation with a circularly polarized light (488 nm, 100 mW/cm<sup>2</sup>) for 0, 30, 60, 90, and 120 min from left to right. The scale bars are 10 μm.

Figure S14a-c show the top-view OM images of the JPs with the IAC-4 parts on the scale of around 11, 7.5, and 5 μm upon irradiation respectively, and the irradiation time is 0, 30, 60, 90, and 120 min from left to right. As demonstrated in the OM images, both the IAC-4 part and the H<sub>2</sub>pdca-PDMS pad remain circular in shape during irradiation for the cases of JPs with different sizes. By comparing the OM images in the same row upon

irradiation for different time periods, the increasing diameter of the H<sub>2</sub>pdca-PDMS pad can be observed to exhibit the flattening deformation of IAC-4 part with increasing irradiation time. Also, the JPs with larger sizes exhibit smaller deformation degree and slower deformation rate, which can be concluded by comparing the OM images in each column. Similar to the case of linearly polarized light irradiation, this size-dependent effect can be ascribed to the absorption screening effect.

The diameters of the IAC-4 dome and the H<sub>2</sub>pdca-PDMS pad can be used to describe the deformation degree, which are respectively defined as  $D_1$  and  $D_2$  and then plotted against the irradiation time as shown in Figure S15. The  $D_1$  and  $D_2$  values are obtained by statistics of 25 JPs with the IAC-4 domes on the scale of around 7.5  $\mu\text{m}$  from the OM images in each case. As shown in the figure,  $D_1$  slightly increases with the irradiation time while  $D_2$  increases to a greater extent especially from 60 to 120 min. The increase in the diameter of the H<sub>2</sub>pdca-PDMS pad together with the flattening of the IAC-4 dome reflect the cooperative deformation behavior of the JPs upon irradiation with a circularly polarized laser beam. Above deformation behavior proves that the interfacial interaction between the two phases of the JPs is strong enough to overcome the stress inside the H<sub>2</sub>pdca-PDMS pad that is adhered on the substrate, as discussed in main text.



**Figure S15.** Variation of the sizes of the IAC-4 part and the H<sub>2</sub>pdca-PDMS part with the irradiation time of the circularly polarized light (488 nm, 100 mW/cm<sup>2</sup>) in the case of

the IAC-4/H<sub>2</sub>pdca-PDMS (w/w = 1/1) JPs with the IAC-4 dome on the scale of 7.5  $\mu\text{m}$ . The definition of D<sub>1</sub> and D<sub>2</sub> is schematically illustrated in the inset.

## References

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