Synthesis and Structure–Property Relationships of Lipoic Acid–Based Conductive Elastomers

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Experimental Section

Materials

Lipoic acid (LA, 99%), 3-Buten-1-ol (BOL, 98%), Dichloromethane (DCM, 99.5%), ethyl acetate (EtOAc, AR), Petroleum ether (PE, AR), Tetrahydrofuran (THF, AR) was purchased from Adamas-beta. N,N'-Methylenebisacrylamide (MBA, 99%), 4-Methoxyphenol (MEHQ, 99%), 1,3-Diisopropenylbenzene (DIB, 97%), 4-Dimethylaminopyridine (DMAP, 99%) was purchased from Aladdin. Allyl glycidyl ether (AGE, 99%), Acryloyl chloride (96%), Magnesium sulfate (MgSO4, AR), Triethylamine (TEA, 99%), 1,10-Diaminodecane (DAD, 97%), Lithium bis((trifluoromethyl)sulfonyl)azanide (LiTFSI, 99.5%), Lithium bromide (LiBr, 99%), was purchased from Macklin. 1-Ethyl-3-(3-dimethyllaminopropyl)carbodiimide hydrochloride (EDC·HCl, 99%) was purchased from Bide Pharmatech. ACAD was synthesis according to the previous literature [1].

Characterization

Fourier transform infrared (FTIR) spectra were recorded using a Nicolet 6700 spectrometer (Thermo Fisher Scientific, USA) at room temperature over the range of 4000–400 cm^{-1}. Raman spectra were collected on a LabRAM HR UV 800 spectrometer (Horiba, France) using a 785 nm excitation laser. Water contact angle measurements were performed using a video-based optical contact angle goniometer (OCA 30, Dataphysics, Germany). A droplet volume of 2 μL was used, and measurements were taken at four different positions on each sample to obtain reliable data. Optical transmittance was measured using a UV–vis spectrophotometer (Lambda 35, PerkinElmer, USA), with the sample thickness fixed at 1 mm.Rheological measurements were carried out on an advanced rotational rheometer (MCR 302, Anton Paar, Austria) at 25 °C with a strain of 0.1% over an angular frequency range of 1–100 rad s^{-1}, and data were collected for 100 s. Differential scanning calorimetry (DSC) was conducted using a STARe SW system (Mettler Toledo, USA) over a temperature range of 25–200 °C at a heating rate of 5 °C min^{-1}. Dynamic mechanical analysis (DMA) was performed on a DMA Q800 instrument (TA Instruments, USA) from −120 to 50 °C at a heating rate of 3 °C min^{-1}. Rectangular specimens with dimensions of 2 mm × 10 mm × 40 mm were used for DMA measurements. Tensile, cyclic tensile, and adhesion tests were carried out at room temperature using a dual-column universal testing machine (Instron 5966, TA Instruments, USA). The surface and fracture morphologies of the materials were characterized by field-emission scanning electron microscopy (Nova NanoSEM, FEI, USA). Fracture surfaces were obtained by brittle fracture after

immersing the samples in liquid nitrogen for 3 min. LabRam HR UV 800 (Horiba, France). ^{1}H-NMR spectra were recorded on an AVANCE400MHz nuclear magnetic resonance spectrometer (Bruker, Germany,) with deuterated chloroform (CDCl3) as the solvent.

Electrical tests

Electrical conductivity was measured using an electrochemical workstation (CHI 760e, Shanghai Chenhua Instruments, China), and each sample was tested three times to ensure reproducibility.The resistance of cylindrical ICEs was measured by electrochemical impedance spectroscopy over a frequency range of 1 to 10^{6} Hz, and the ionic conductivity was calculated according to the following equation (1).

$$\sigma = \frac{L}{R \times S} \#(1)$$

Here, L, R, and S represent the height, resistance, and sectional area of the sample, respectively.

Mechanical tests

Tensile and cyclic tensile specimens were cut into dog-bone ‑ shaped samples with a total length of 35 mm, a gauge length of 15 mm, a width of 2 mm, and a thickness of 1 mm. All tensile and cyclic tests were conducted at a constant crosshead speed of 50 mm min^{-1}. The tensile strength was calculated as the ratio of the maximum force at fracture to the initial cross-sectional area of the specimen (2). The elongation at break was defined as the ratio of the displacement at fracture to the initial gauge length (3) and was calculated according to the following equations:

$$\sigma = F_{max}/A \#(2)$$

$$\varepsilon_b = L_b/L_0 \times 100\% \#(3)$$

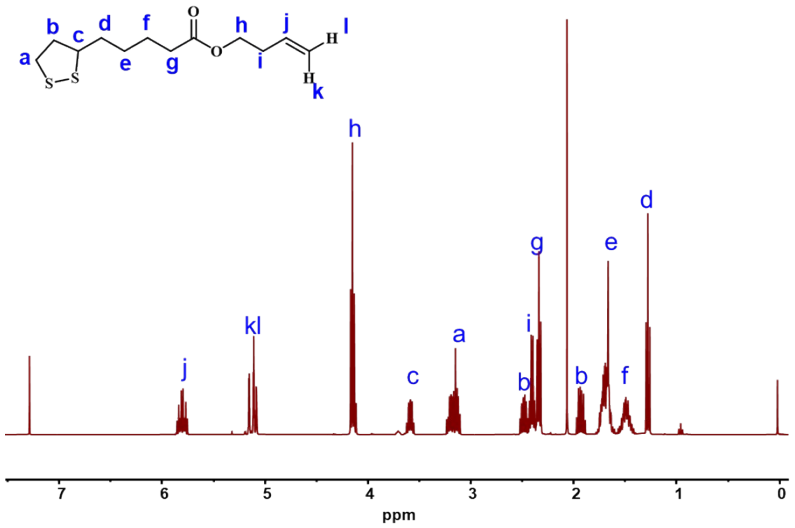

Figure S1: 1H-NMR spectra of LA-BOL

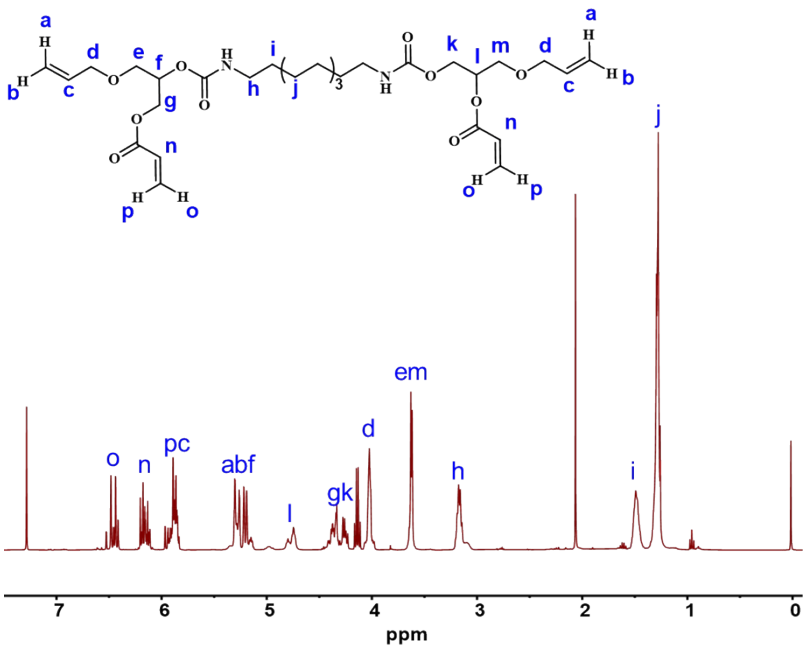

Figure S2: 1H-NMR spectra of ACAD

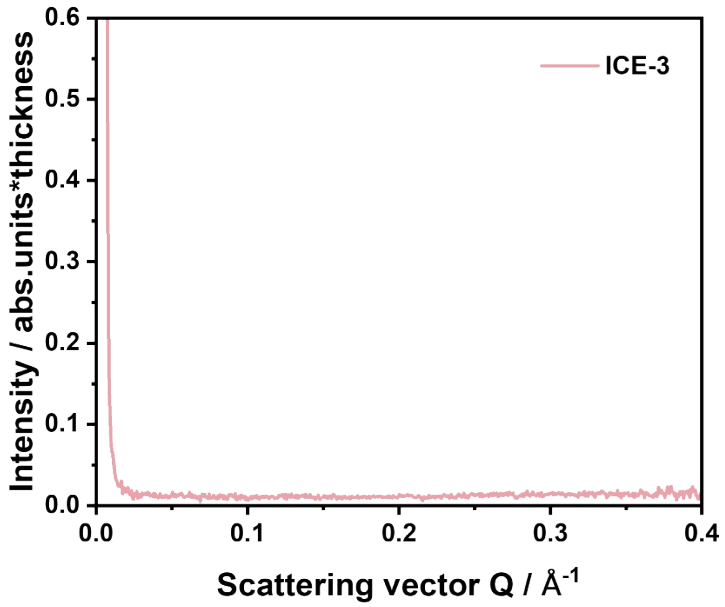

Figure S3: SAXS of ICE-3

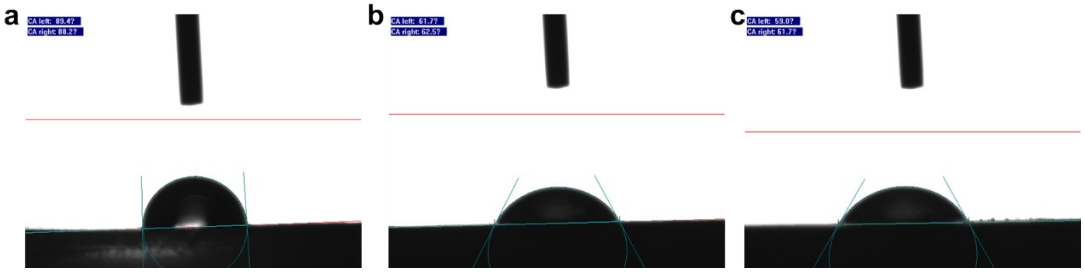

Figure S4: Water contact angle images of ICE-3 (a), ICE-6 (b), and ICE-9 (c) obtained using a 2 µL water droplet.

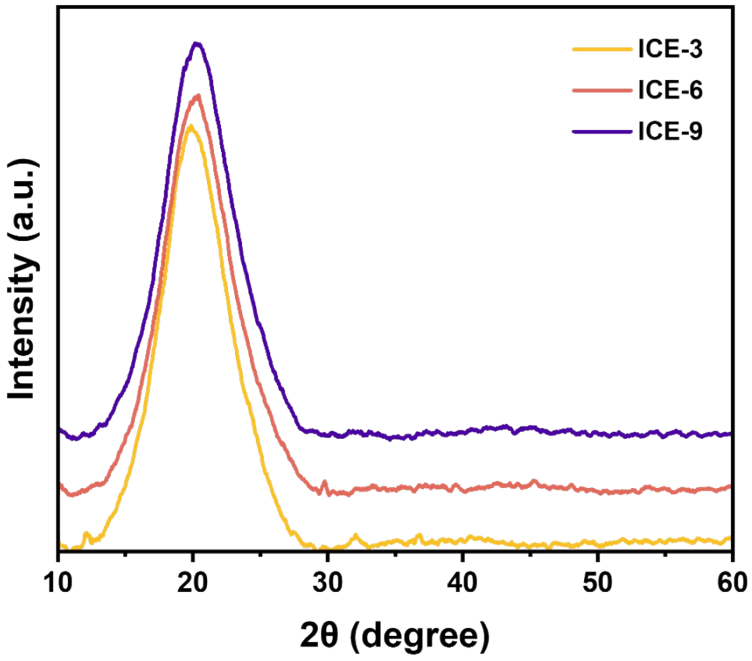

Figure S5: XRD of ICE-3, ICE-6, ICE-9

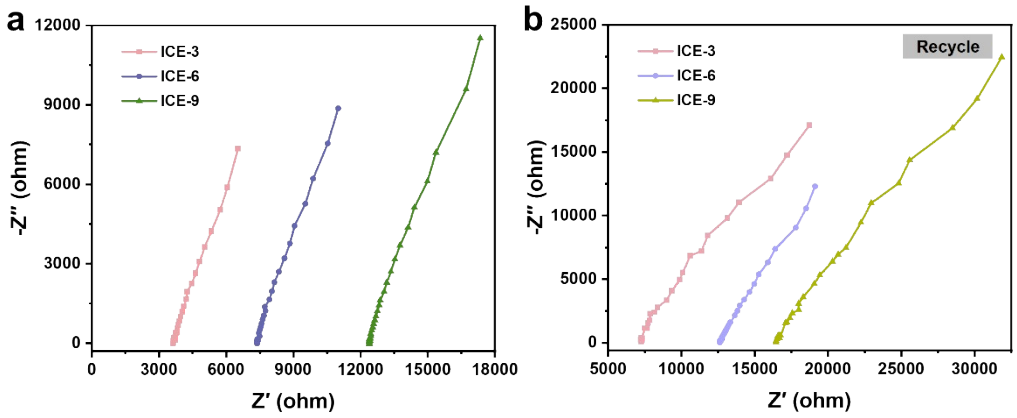

Figure S6: Electrochemical impedance spectroscopy (EIS) plots of ICE-3, ICE-6, ICE-9 and their corresponding recycled samples (25 ℃)

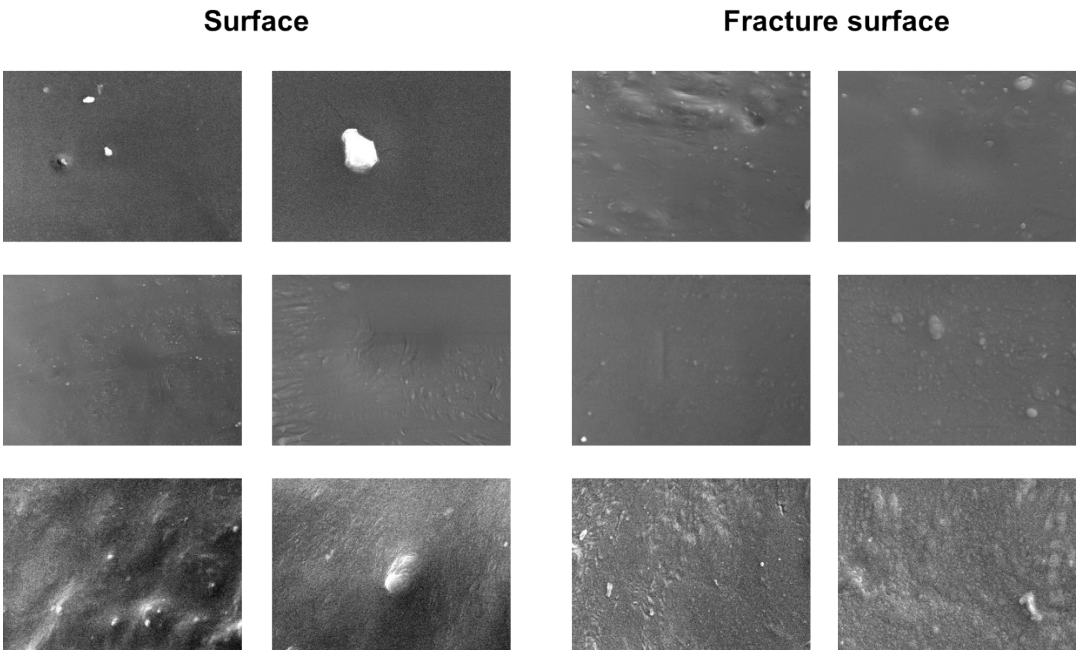

Figure S7: (a,e,i) Surface morphologies of ICE-3, ICE-6, and ICE-9 at ×1000 magnification. (b,f,j) Corresponding locally magnified surface images at ×5000 magnification. (c,g,k) Cross-sectional morphologies of ICE-3, ICE-6, and ICE-9 at ×1000 magnification. (d,h,i) Corresponding locally magnified cross-sectional images at ×5000 magnification.

References

[1] W. Xu, T. Shen, Y. Ding, H. Ye, B. Wu, F. Chen, Wearable and Recyclable Water-Toleration Sensor Derived from Lipoic Acid, Small 20 (2024) 2310072. https://doi.org/10.1002/smll.202310072.