Supporting Information for

Synthesis of Poly(ethylene-*co*-acrylic acid) *via* a Tandem Hydrocarboxylation/Hydrogenation of Poly(butadiene)

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General Considerations. Unless otherwise noted, all experiments were performed in a nitrogen purged glove box or under an atmosphere of nitrogen using standard Schlenk techniques. N,N'dimesityl-4.6-diketo-5,5-dimethylpyrimidin-2-ylidene (1) was prepared according to a literature procedure.¹ All other reagents were commercially available and used as received. Unless otherwise noted, solvents were dried over 3 Å molecular sieves or using a Vacuum Atmospheres Company solvent purification system, and then subsequently stored over 3 Å molecular sieves. ¹H and ¹³C NMR data were collected on a Varian Unity INOVA 400 MHz spectrometer. Chemical shifts (δ) are reported in ppm and referenced downfield from (CH₃)₄Si using the residual solvent peak as an internal standard (¹H: C₆D₆, 7.15 ppm; ¹³C: C₆D₆ 128.0 ppm). IR spectra were recorded using a Thermo Scientific Nicolet iS5 system equipped with an iD3 attenuated total reflectance (ATR) attachment (germanium crystal). Gel permeation chromatography (GPC) was performed on a Viscotek HPLC system consisting of two Viscotek I-series columns (1 \times MBHMW-3078 and 1 \times MBMMW-3078) arranged in series and thermostated to 24 °C, a Viscotek 2001 GPCmax solvent/sample module, a Viscotek 270 dual detector, and a Viscotek 3580 refractive index (RI) detector. Number average molecular weight (M_n) and polydispersity (PDI) data determined by GPC are reported relative to polystyrene standards in tetrahydrofuran (THF) or as the absolute molecular weights (determined using triple detection). HPLC-MS was performed on an Agilent 1200 Series HPLC equipped with a Gemini C18 5 micron 2 \times 50 mm column with 2 \times 4 mm guard (eluent = gradient ramp 5–95% of 0.1%) formic acid in water into 0.1% formic acid in acetonitrile), UV-vis analysis was conducted at 254 nm and mass spectrometry was performed with an Agilent 6130 single quadrupole mass spectrometer (ionization = atmospheric-pressure chemical ionization in positive mode). Elemental analyses were performed by Midwest Microlab, LLC (Indianapolis, IN). Thermogravimetric analysis (TGA) and differential scanning calorimetry (DSC) were performed using a Mettler-Toledo TGA/SDTA851e and DSC823e, respectively, under an atmosphere of nitrogen at a temperature scan rate of 10 °C min⁻¹. The decomposition temperature (T_d) was defined as the temperature at which 10% weight loss was observed. Percent crystallinity (γ) was determined by DSC assuming a melt enthalpy of 293 J g⁻¹ for 100% crystalline poly(ethylene).² Dynamic mechanical analyses were performed using a TA Instruments O800. The storage moduli were determined in the tensile mode from -150 to 150 °C at a ramp rate of 3 °C min⁻¹ (the temperature was held at -150 °C for 5 min before starting the experiment), with a preload force of 0.01 N, an amplitude of 15 µm, and a frequency of 1 Hz. Stress strain curves were acquired in the tensile mode at 30 °C with a preload force of 0.001 N and a force ramp rate of 1.0 N min⁻¹.

Synthesis of poly(butadiene) cyclopropanated with the DAC (pBD-DAC) containing 30 mol% cyclopropyl groups. An oven dried 8 mL vial was charged with *cis*-poly(butadiene) (25 mg, 0.46 mmol) and minimal toluene (0.5 mL). The resulting mixture was stirred until the dissolution of *cis*-poly(butadiene) was complete (2 h), after which 2 equiv. of 1 (346 mg, 0.92 mmol) per butadiene repeat unit was added. The vial was re-sealed, and the resulting reaction mixture was stirred at 60 °C. After 36 h, the reaction mixture was cooled to 25 °C, exposed to air, and precipitated into methanol (10 mL). Isolation of the product by filtration and drying under high vacuum afforded the desired polymer as a colorless solid (47 mg, 0.28 mmol) in 61% yield based on 30 mol% cyclopropanation. ¹H NMR (C₆D₆, 400.27 MHz): δ 0.27 (s, 2H), 0.44 (s, 6H), 1.81–2.62 (br m, 27.3 H), 5.50 (s, 4.7H), 6.64–6.78 (m, 4H). ¹³C NMR (C₆D₆, 100.49 MHz): δ 19.82, 20.98, 25.08, 27.89, 32.12, 34.56, 46.85, 49.02, 99.88, 129.20, 129.56, 129.79, 135.03,

135.50, 138.60, 170.98. IR (ATR): 3006, 2918, 2850, 1684, 1658, 2608, 1579, 1422, 1405, 1354, 1261, 1102, 1030, 734. GPC (polystyrene equivalent): $M_n = 54$ kDa; PDI = 1.7. GPC (absolute): $M_n = 487$ kDa; theoretical MW = 507 kDa.

Synthesis of poly(butadiene-*co*-(ethylene-*alt*-acrylic acid)) (pBDEAA) (15 mol% AA). An oven dried 8 mL vial was charged with pBD-DAC (30 mol% cyclopropyl groups) (47 mg, 0.28 mmol), toluene (1.5 mL), concentrated HCl acid (0.5 mL) and glacial acetic acid (0.5 mL). The vial was sealed, and the resulting mixture was stirred at 80 °C. After 24 h, the reaction mixture was cooled to 25 °C, exposed to air, and precipitated into methanol (10 mL). The resulting polymeric material was dissolved in minimal dichloromethane (0.5 mL) and precipitated into fresh methanol (15 mL). Isolation by filtration and drying under high vacuum afforded the desired polymer as a pale yellow solid (16 mg, 0.24 mmol) in 84% yield based on a 15 mol% acrylic acid content. ¹H NMR (C₆D₆, 400.27 MHz): δ 1.35 (s, 2H), 1.50 (s, 4H), 2.11–2.19 (m, 18.6H), 2.29 (m, 1H), 5.49–5.51 (m, 9.3H). ¹³C NMR (C₆D₆, 100.49 MHz): δ 26.98, 29.89, 30.98, 34.45, 46.56, 129.79, 172.45. IR (ATR): 3006, 2917, 2849, 1705, 1677, 1452, 1433, 1262, 1099, 1025, 911, 737. GPC (polystyrene equivalent): M_n = 110 kDa; PDI = 1.5. GPC (absolute): M_n = 198 kDa; theoretical MW = 206 kDa.

Synthesis of pBDEAA in a single reaction vessel. An oven dried 8 mL vial was charged with *cis*-poly(butadiene) (48 mg, 0.88 mmol) and minimal toluene (0.5 mL). The resulting mixture was stirred until the dissolution of *cis*-poly(butadiene) was complete (2 h), after which 2 equiv. of **1** (500 mg, 1.33 mmol) per butadiene repeat unit was added. The vial was re-sealed and the resulting reaction mixture was stirred at 60 °C. After 36 h, the reaction mixture was cooled to 25 °C, exposed to air, and toluene (1.0 mL), concentrated HCl acid (0.5 mL) and glacial acetic acid (0.5 mL) were added. The vial was re-sealed, and the resulting mixture was stirred at 80 °C. After 24 h, the reaction mixture was cooled to 25 °C, exposed to air, and precipitated into methanol (15 mL). The resulting polymeric material was dissolved in minimal dichloromethane (0.5 mL) and precipitated into fresh methanol (15 mL). Isolation by filtration and drying under high vacuum afforded the desired polymer as a pale yellow solid (48 mg, 0.73 mmol) in 83% yield based on a 15 mol% acrylic acid content. Spectral data were consistent with the previously described values. GPC (polystyrene equivalent): $M_n = 110$ kDa; PDI = 1.6. GPC (absolute): $M_n = 195$ kDa; theoretical MW = 206 kDa.

Synthesis of poly(ethylene-*co*-acrylic acid) (pEAA) (15 mol% AA). A solution of pBDEAA (15 mol% AA) (100 mg, 1.5 mmol) in toluene (10 mL), and *n*-butanol (5 mL) was added to an oven dried 50 mL round bottom flask. The resulting solution was then degassed by bubbling a nitrogen purge through the stirred solution for 30 min. Solid Wilkinson's catalyst (1.7 mg, 1.8 μ mol) [RhCl(PPh₃)₃] was added to the solution under a positive pressure of nitrogen. The flask was then fitted with a vacuum adapter and sealed in a Parr reactor. The reactor was filled to 400 psi hydrogen gas and purged while stirring (3×), filled to 400 psi hydrogen, sealed, and heated to 80 °C. After 48 h, the reaction mixture was cooled to 25 °C, and the pressure was slowly released from the Parr reactor. The crude reaction solution was precipitated into methanol (100 mL). Isolation by filtration and drying under high vacuum afforded the desired polymer as a pale grey solid (96 mg, 1.4 mmol) in 90% yield. IR (ATR): 2918, 2850, 1702, 1684, 1260, 1089, 1020, 964, 800, 720. Anal. Found: C, 74.95; H, 11.74; O, 13.31.

Synthesis of poly(butadiene-*co*-(ethylene-*alt*-methyl acrylate)) (pBDEMA) (15 mol% MA). A solution of pBDEAA (15 mol% AA) (49 mg, 0.65 mmol) in toluene (4 mL), and methanol (1 mL) was added to an oven dried 25 mL round bottom flask. The resulting solution was then degassed by bubbling a nitrogen purge through the stirred solution for 30 min. Under a positive pressure on nitrogen, a 2.0 M solution of trimethylsilyldiazomethane (0.5 mL, 1.0 mmol) in ether was added and the resulting solution was stirred under a nitrogen atmosphere at 25 °C. After 36 h, the crude reaction solution was precipitated into methanol (20 mL). Isolation by filtration and drying under high vacuum afforded the desired polymer as a yellow solid (41 mg, 0.51 mmol) in 82% yield based on a 15 mol% methyl acrylate content. ¹H NMR (C₆D₆, 400.27 MHz): δ 1.39 (s, 2H), 1.72 (s, 4H), 2.14–2.20 (m, 18.6H), 2.39 (m, 1H), 3.66 (3H), 5.34–5.39 (m, 9.3H). ¹³C NMR (C₆D₆, 100.49 MHz): δ 27.83, 29.45, 30.18, 51.34, 129.90, 169.21. IR (ATR): 3006, 2920, 2816, 1735, 1708, 1423, 1406, 1261, 1240, 1215, 1102, 966, 852, 737. GPC (polystyrene equivalent): M_n = 125 kDa; PDI = 1.7. GPC (absolute): M_n = 215 kDa; theoretical MW = 218 kDa.

Synthesis of poly(ethylene-co-methyl acrylate) (pEMA) (15 mol% MA). A solution of pBDEMA (15 mol% MA) (119 mg, 1.5 mmol) in toluene (12 mL), and *n*-butanol (6 mL) was added to an oven dried 50 mL round bottom flask. The resulting solution was then degassed by bubbling a nitrogen purge through the stirred solution for 30 min. Solid Wilkinson's catalyst (1.4 mg, 1.5 µmol) was added to the solution under a positive pressure of nitrogen. The flask was then fitted with a vacuum adapter and sealed in a Parr reactor. The reactor was filled to 400 psi hydrogen gas and purged while stirring (3x), filled to 400 psi hydrogen, sealed, and heated to 80 °C. After 48 h, the reaction mixture was cooled to 25 °C, and the pressure was slowly released from the Parr reactor. The crude reaction solution was precipitated into methanol (100 mL). Isolation by filtration and drying under high vacuum afforded the desired polymer as a tan solid (106 mg, 1.3 mmol) in 88% yield based on a 15 mol% methyl acrylate content. Spectral data were consistent with literature values.^{3 1}H NMR (C₆D₆, 400.27 MHz): δ 1.27–1.65 (br m, 24.67H), 2.27 (br m, 1H), 3.61 (s, 3H). ¹³C NMR (C₆D₆, 100.49 MHz): δ 27.83, 29.45, 32.18, 51.23, 46.21, 169.89. IR (ATR): 2919, 2850, 1736, 1710, 1463, 1421, 1260, 1099, 1017, 851, 799. GPC (polystyrene equivalent): $M_n = 111 \text{ kDa}$; PDI = 1.6. GPC (absolute): $M_n = 224 \text{ kDa}$; theoretical MW = 219 kDa.

Synthesis of poly(butadiene) cyclopropanated with DAC (pBD-DAC) containing 12 mol% cyclopropyl groups. An oven dried 8 mL vial was charged with *cis*-poly(butadiene) (25 mg, 0.46 mmol) and minimal toluene (0.5 mL). The resulting mixture was stirred until the dissolution of *cis*-poly(butadiene) was complete (2 h), after which 1 equiv. of 1 (174 mg, 0.46 mmol) per butadiene repeat unit was added. The vial was re-sealed, and the resulting reaction mixture was stirred at 60 °C. After 36 h, the reaction mixture was cooled to 25 °C, exposed to air, and precipitated into methanol (10 mL). Isolation of the product by filtration and drying under high vacuum afforded the desired polymer as a colorless solid (41 mg, 0.38 mmol) in 84% yield based on 12 mol% cyclopropanation. ¹H NMR (C₆D₆, 400.27 MHz): δ 0.27 (s, 2H), 0.44 (s, 6H), 1.81–2.62 (br m, 51.3 H), 5.50 (s, 14.7H), 6.64–6.78 (m, 4H). ¹³C NMR (C₆D₆, 100.49 MHz): δ 19.82, 20.98, 25.08, 27.89, 32.12, 34.56, 46.85, 49.02, 99.88, 129.20, 129.56, 129.79, 135.03, 135.50, 138.60, 170.98. IR (ATR): 3006, 2918, 2850, 1684, 1658, 1579, 1422, 1405, 1354, 1261, 1102, 1030, 734. GPC (polystyrene equivalent): M_n = 70 kDa; PDI = 1.9. GPC (absolute): M_n = 282 kDa; theoretical MW = 299 kDa. T_d = 345 °C; T_g = -75 °C.

Synthesis of poly(butadiene-*co*-(ethylene-*alt*-acrylic acid)) (pBDEAA) (6 mol% AA). An oven dried 8 mL vial was charged with pBD-DAC (12 mol% cyclopropyl groups) (41 mg, 0.38 mmol), toluene (1.5 mL), concentrated HCl acid (0.5 mL) and glacial acetic acid (0.5 mL). The vial was sealed, and the resulting mixture was stirred at 80 °C. After 24 h, the reaction mixture was cooled to 25 °C, exposed to air, and precipitated into methanol (10 mL). The resulting polymeric material was dissolved in minimal dichloromethane (0.5 mL) and precipitated into fresh methanol (15 mL). Isolation by filtration and drying under high vacuum afforded the desired polymer as a pale yellow solid (23 mg, 0.35 mmol) in 92% yield based on a 6 mol% acrylic acid content. ¹H NMR (C₆D₆, 400.27 MHz): δ 1.35 (s, 2H), 1.50 (s, 4H), 2.11–2.19 (m, 59.8H), 2.29 (m, 1H), 5.49–5.51 (m, 29.9H). ¹³C NMR (C₆D₆, 100.49 MHz): δ 26.98, 29.89, 30.98, 34.45, 46.56, 129.79, 172.45. IR (ATR): 3006, 2917, 2849, 1705, 1677, 1452, 1433, 1262, 1099, 1025, 911, 737. GPC (polystyrene equivalent): M_n = 89 kDa; PDI = 1.7. GPC (absolute): M_n = 185 kDa; theoretical MW = 187 kDa. T_d = 392 °C; T_g = -105 °C.

Synthesis of poly(ethylene-*co*-acrylic acid) (pEAA) (6 mol% AA). A solution of pBDEAA (6 mol% AA) (127 mg, 1.9 mmol) in toluene (12 mL), and *n*-butanol (6 mL) was added to an oven dried 50 mL round bottom flask. The resulting solution was then degassed by bubbling a nitrogen purge through the stirred solution for 30 min. Solid Wilkinson's catalyst (1.7 mg, 1.8 μ mol) [RhCl(PPh₃)₃] was added to the solution under a positive pressure of nitrogen. The flask was then fitted with a vacuum adapter and sealed in a Parr reactor. The reactor was filled to 400 psi hydrogen gas and purged while stirring (3×), filled to 400 psi hydrogen, sealed, and heated to 80 °C. After 48 h, the reaction mixture was cooled to 25 °C, and the pressure was slowly released from the Parr reactor. The crude reaction solution was precipitated into methanol (100 mL). Isolation by filtration and drying under high vacuum afforded the desired polymer as a pale grey solid (112 mg, 1.7 mmol) in 88% yield. IR (ATR): 2918, 2850, 1702, 1684, 1260, 1089, 1020, 964, 800, 720. Anal. Found: C, 80.42; H, 13.11; O, 6.51.

Synthesis of hydrogenated *cis*-poly(butadiene) (H₂-pBD). A solution of *cis*-pBD (500 mg, 9.2 mmol) in toluene (50 mL) and *n*-butanol (25 mL) was added to an oven dried 50 mL round bottom flask. The resulting solution was then degassed by bubbling a nitrogen purge through the stirred solution for 30 min. Solid Wilkinson's catalyst (85 mg, 9.0 μ mol) was added to the solution under a positive pressure of nitrogen. The flask was then fitted with a vacuum adapter and sealed in a Parr reactor. The reactor was filled to 400 psi hydrogen gas and purged while stirring (3×), filled to 400 psi hydrogen, sealed, and heated to 80 °C. After 48 h, the reaction mixture was cooled to 25 °C, and the pressure was slowly released from the Parr reactor. The crude reaction solution was precipitated into methanol (200 mL). Isolation by filtration and drying under high vacuum afforded the desired polymer as a tan solid (513 mg, 9.1 mmol) in 99% yield. IR (ATR): 2917, 2849, 1472, 1463, 1098, 965, 803, 730, 719.

Ozonolysis of pBD-DAC. Using a modified literature procedure,⁴ a solution of pBD-DAC (30 mol% cyclopropyl groups) (283 mg, 5.2 mmol) in CD₂Cl₂ (25 mL) was added to a 100 mL round bottom flask and the resulting solution was cooled to -78 °C in an acetone/dry ice bath. After equilibrating at -78 °C (15 min), a steady stream (rate = 3 mL min⁻¹) of ozone was bubbled through the solution while a 1 M solution of Sudan III in methanol (50 µL) was added to the reaction as an indicator. After 1 h, the pink indicator was consumed and the reaction was colorless. The atmosphere of the flask was then changed from ozone to nitrogen and dimethyl

sulfide (2 mL, 27.2 mmol) was added to the solution at -78 °C. The solution was stirred and slowly warmed to 25 °C under an atmosphere of nitrogen. After 16 h at 25 °C, the resulting crude mixture was examined by NMR spectroscopy (CD₂Cl₂; see main text) and HPLC-MS (see Table S1).

Table S1. Summary of the data obtained from the HPLC-MS analysis of the pBD-DAC (containing 30 mol% cyclopropyl groups) subjected to ozonolysis.^a

retention time ^a	area $(\%)^b$	mass ^c	product
4.83	99.43	517	2 •H⁺
8.70	0.12	377	$1 \bullet \mathrm{H}^+$
8.88	0.25	496	4 •H⁺
10.49	0.20	948	3 •H⁺

^{*a*} Ozonolysis conditions: 1) CD₂Cl₂, O₃ (rate = 3 mL min⁻¹), -78 °C; 2) Me₂S (5 equiv.), -78 to 25 °C, 16 h. ^{*b*} HPLC eluent: 5–95 % of 0.1 % formic acid in water into 0.1 % formic acid in CH₃CN via a gradient ramp. ^{*c*} Mass spectrometry was performed in the positive mode using atmospheric pressure chemical ionization.



Figure S1. IR spectrum of pBD-DAC (30 mol% cyclopropyl groups) (ATR).



Figure S2. IR spectra of **pBDEMA** (15 mol% MA) (black) and **pEMA** (15 mol% MA) (red) (ATR).



Figure S3. IR spectra of H₂-pBD (ATR).



Figure S4. IR spectra of **pBD-DAC** (12 mol% cyclopropyl groups) (black), **pBDEAA** (6 mol% AA) (blue) and **pEAA** (6 mol% AA) (red) (ATR).



Figure S5. Storage modulus versus temperature for pEAA (15 mol% AA) (black), pEAA (6 mol% AA) (blue) and H₂-pBD (red). Data were acquired via DMA in the tension mode at a ramp rate of 3 °C min⁻¹.



Figure S6. Stress strain curves acquired for **pEAA** (15 mol% AA) (five separate runs indicated). Data were acquired via DMA in the tension mode at 30 $^{\circ}$ C with a force ramp rate of 1.0 N min⁻¹.



Figure S7. Stress strain curves acquired for **pEAA** (6 mol% AA) (five separate runs indicated). Data were acquired via DMA in the tension mode at 30 °C with a force ramp rate of 1.0 N min^{-1} .



Figure S8. Stress strain curves acquired for H_2 -pBD (five separate runs indicated). Data were acquired via DMA in the tension mode at 30 °C with a force ramp rate of 1.0 N min⁻¹.



Figure S9. ¹H NMR spectrum of pBD-DAC (30 mol% cyclopropyl groups) in C_6D_6 .



Figure S10. ¹³C NMR spectrum of pBD-DAC (30 mol% cyclopropyl groups) in C_6D_6 .



Figure S11. ¹H NMR spectrum of pBDEAA (15 mol% AA) in C₆D₆.



Figure S12. ¹³C NMR spectrum of pBDEAA (15 mol% AA) in C_6D_6 .



Figure S13. ¹H NMR spectrum of pBDEMA (15 mol% MA) in C₆D₆.



Figure S14. ¹³C NMR spectrum of pBDEMA (15 mol% MA) in C_6D_6 .



Figure S15. ¹H NMR spectrum of pEMA (15 mol% MA) in C₆D₆.



Figure S16. ¹³C NMR spectrum of pEMA (15 mol% MA) in C_6D_6 .



Figure S17. ¹H NMR spectrum of pBD-DAC (12 mol% cyclopropyl groups) in C₆D₆.



Figure S18. ¹³C NMR spectrum of pBD-DAC (12 mol% cyclopropyl groups) in C_6D_6 .



Figure S19. ¹H NMR spectrum of pBDEAA (6 mol% AA) in C₆D₆.



Figure S20. ¹³C NMR spectrum of pBDEAA (6 mol% AA) in C₆D₆.

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