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Polymerization–cyclodepolymerization of polypropylene carbonate mediated by cobalt catalyst

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Experimental part

Polymer synthesis

All air or water sensitive reactions were carried out under dry nitrogen using drybox or standard Schlenk-line techniques. *rac*-(salcy)CoOC(O)C_6F_5 and PPNCl were prepared as previously described[S1,S2]. *rac*-PO was dried over CaH_2, distilled under argon atmosphere and vacuum transferred before use. The mixture of *rac*-(salcy)CoO C(O)C_6 and PPNCl was dissolved in *rac*-PO (6 ml) in a 10 ml vial equipped with magnetic stir bar. The mixture was allowed to stir until red-brown homogeneous solution was formed and then the vial was placed into a pre-dried 100 ml autoclave, which was pressurized to appropriate pressure with CO_2 and left on stirring for 64 h. After that, the mixture was dissolved in 50 ml CH_2Cl_2, concentrated in vacuum and precipitated from CH_2Cl_2/MeOH (10:1, v/v) mixture into Et_2O. The resulting polymer was dried in vacuum to a constant weight.

Methods

FTIR spectra in ATR mode (diamond crystal) of the polymer films were recorded using Spectrum Two FT-IR Spectrometer (PerkinElmer) in the range of 4000–600 cm^{-1}. NMR spectra were obtained on a Bruker Avance III HD (400 MHz ^1H, 101 MHz ^{13}C) in $CDCl_3$. The SEC measurements were performed in THF at 40 °C with a flow rate of 1.0 mL/min using a 1260 Infinity II GPC/SEC Multidetector System chromatograph (Agilent, Santa Clara, CA, USA) equipped with two PLgel 5 μm MIXED B columns. The SEC system was calibrated using narrow dispersed linear poly(methyl methacrylate) standards with MW ranging from 0.8 to 2000 kDa.

Differential scanning calorimetry was performed on a Netzsch DSC 204 (Netzsch, Germany) in the atmosphere of dry gas (air, argon) at flow rate of 100 mL/min and heating rate 10 ^{o}C/min in argon.

Thermogravimetric analysis (TGA) was performed using Mettler TA4000 system at a heating rate 10 ^{o}C/min.

Hot pressing was carried out on an individually designed thermal press at 130 ^{o}C and a pressure of 18 MPa for 10 minutes, followed by cooling by an air flow with a temperature of 20 ^{o}C.

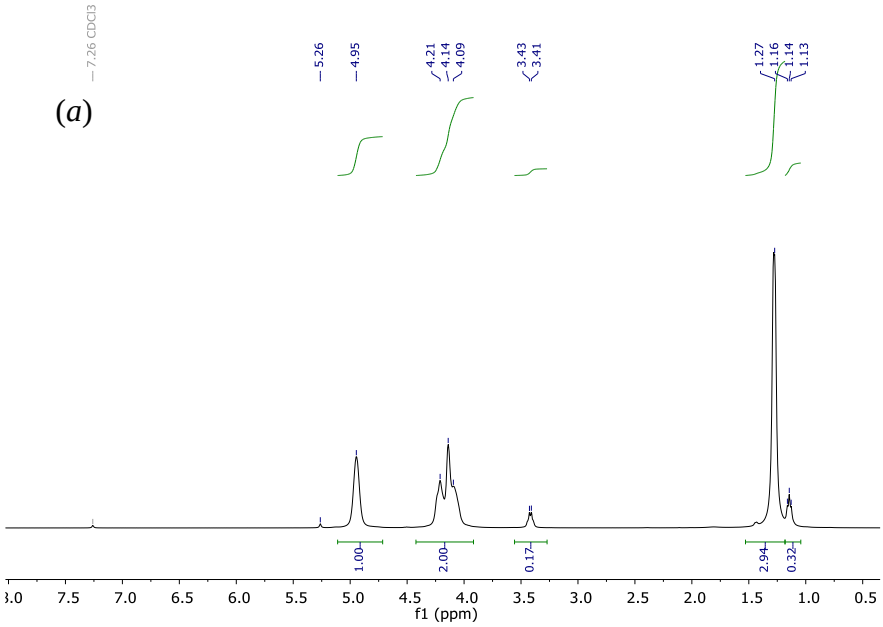
Ashing was carried out using muffle furnace P330 (Nabertherm) followed by the study of elemental composition of the resulting mixture by ICP-MS. ICP-MS was carried out on an Elan DRC-e device (Perkin-Elmer) with the following parameters: operating frequency of the ICP generator 40 MHz; output power 1.1 kW; plasma-forming argon flow 15 L/min; argon transport flow 0.90 L/min; argon cooling flow 0.5 L/min; sample flow rate 0.85 mL/min.

References

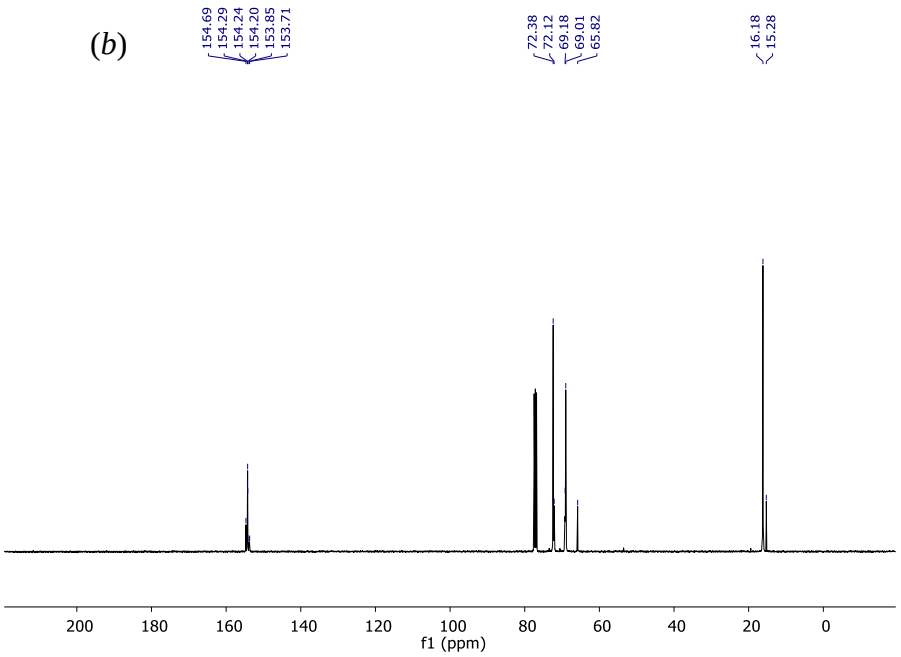
S1. C. T. Cohen, T. Chu and G. W. Coates, *J. Am. Chem. Soc*., 2005, **127**, 10869; https://doi.org/10.1021/ja051744l.

S2. V. Yu. Kukushkin and A. I. Moiseev, *Inorg. Chim. Acta*, 1990, **176**, 79; https://doi.org/10.1016/S0020-1693(00)85095-1.

(*a*)


7.26 CDCl3
5.26
4.95
4.21
4.14
4.09
3.43
3.41
1.27
1.16
1.14
1.13
1.00
2.00
0.17
2.94
0.32
f1 (ppm)

(*b*)


154.69
154.29
154.24
154.20
153.85
153.71
72.38
72.12
69.18
69.01
65.82
16.18
15.28
f1 (ppm)

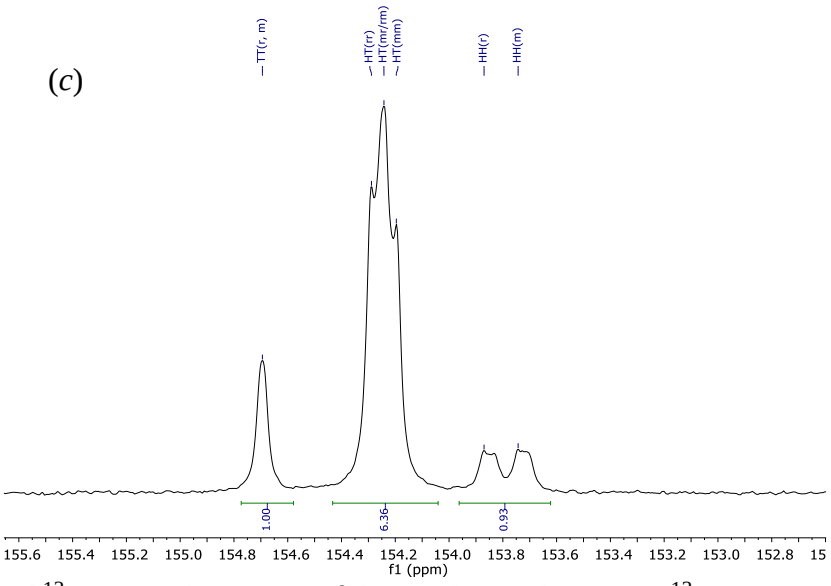
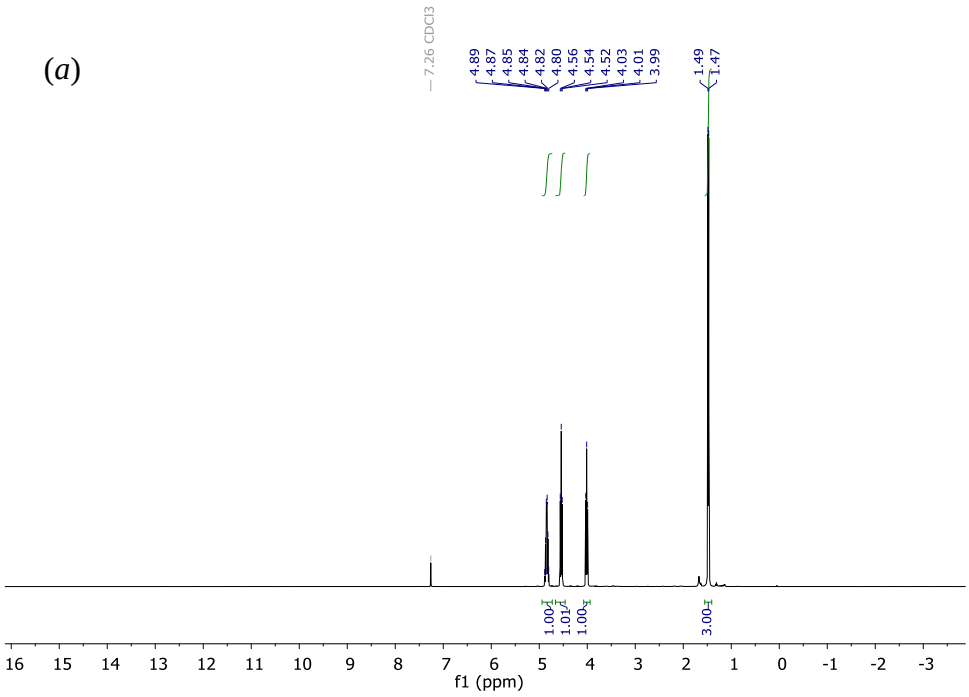


Figure S1 ^{1}H (*a*) and ^{13}C NMR (*b*) spectra of the synthesized PPC; (*c*) ^{13}C NMR spectrum of the PPC in the range of 152 – 156 ppm.



(*b*)

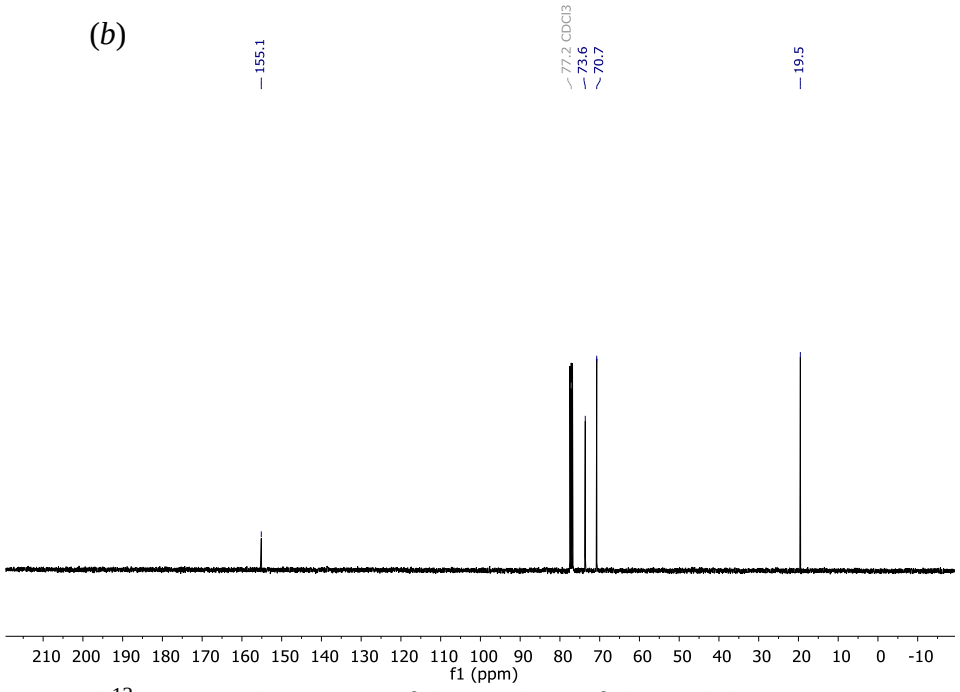


Figure S2 ^{1}H (*a*) and ^{13}C NMR (*b*) spectra of the product of thermal decomposition at 120 ^{o}C during 144 h.

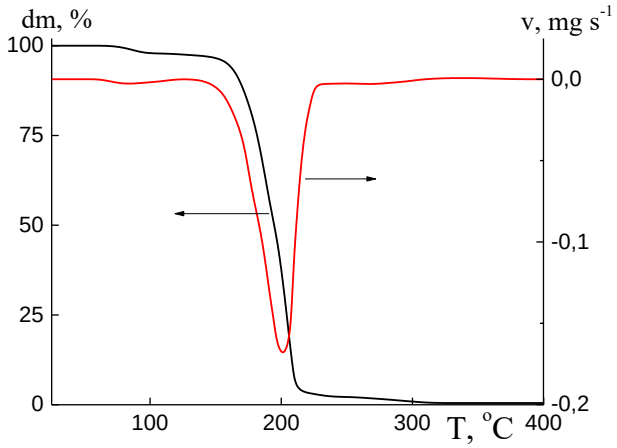


Figure S3 TGA and DTC curves of the synthesized PPC recorded at a heating rate 10 ^{o}C/min