Morphology and thermal properties of poly(L-lactic acid) nucleated with 2,2'-(butane-1,4-diylbis(oxy))di(benzohydrazide)

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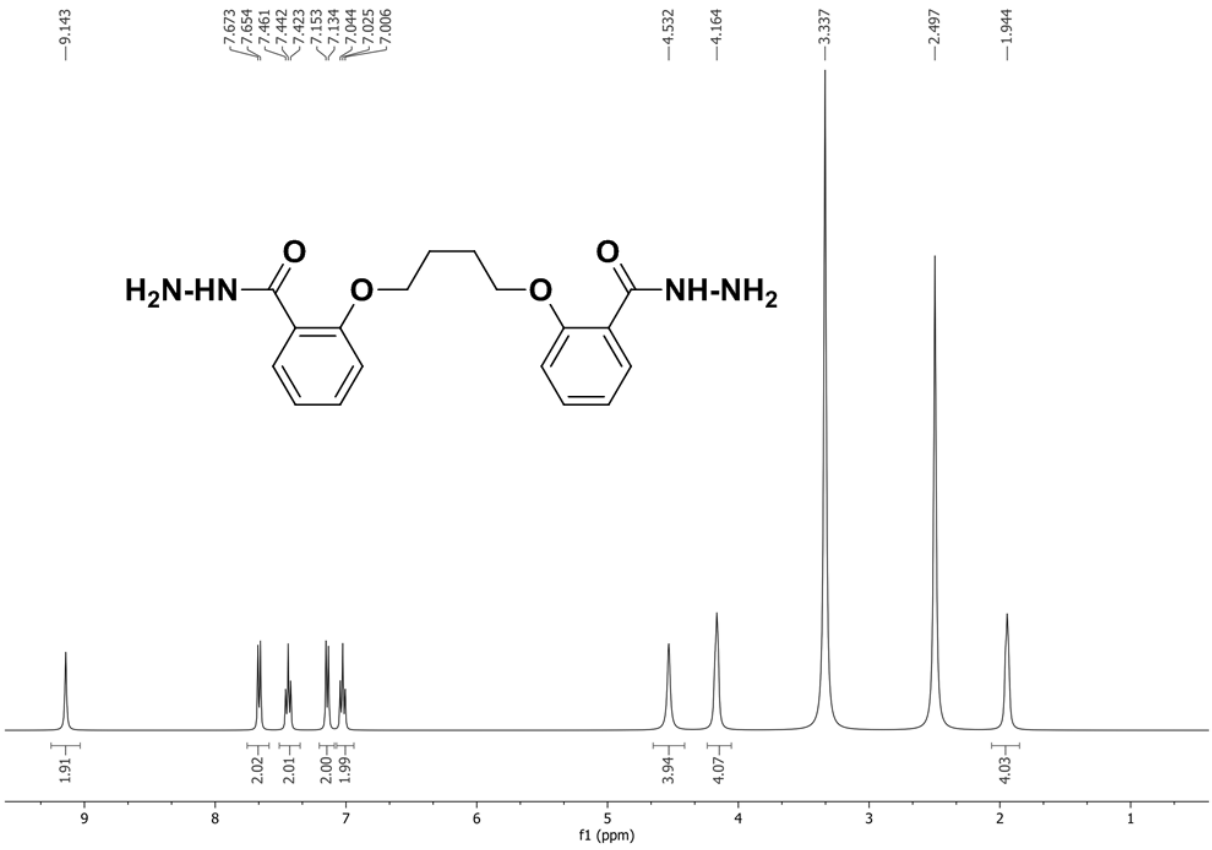
Experimental

All solvents were acquired from commercial sources and used as received unless otherwise stated. All other chemicals were acquired from Merck or Aldrich. These chemicals were used without further purification. The melting points were measured on a Stuart melting point apparatus and are uncorrected. NMR spectra were recorded on a Bruker Avance III 400 spectrometer (400 MHz for ^{1}H-NMR and 100 MHz for ^{13}C-NMR) using TMS as an internal standard and DMSO-d_6 as solvent and chemical shifts were expressed as δ ppm units. Elemental analyses were carried out on a EuroVector instrument C, H, N analyzer EA3000 Series.

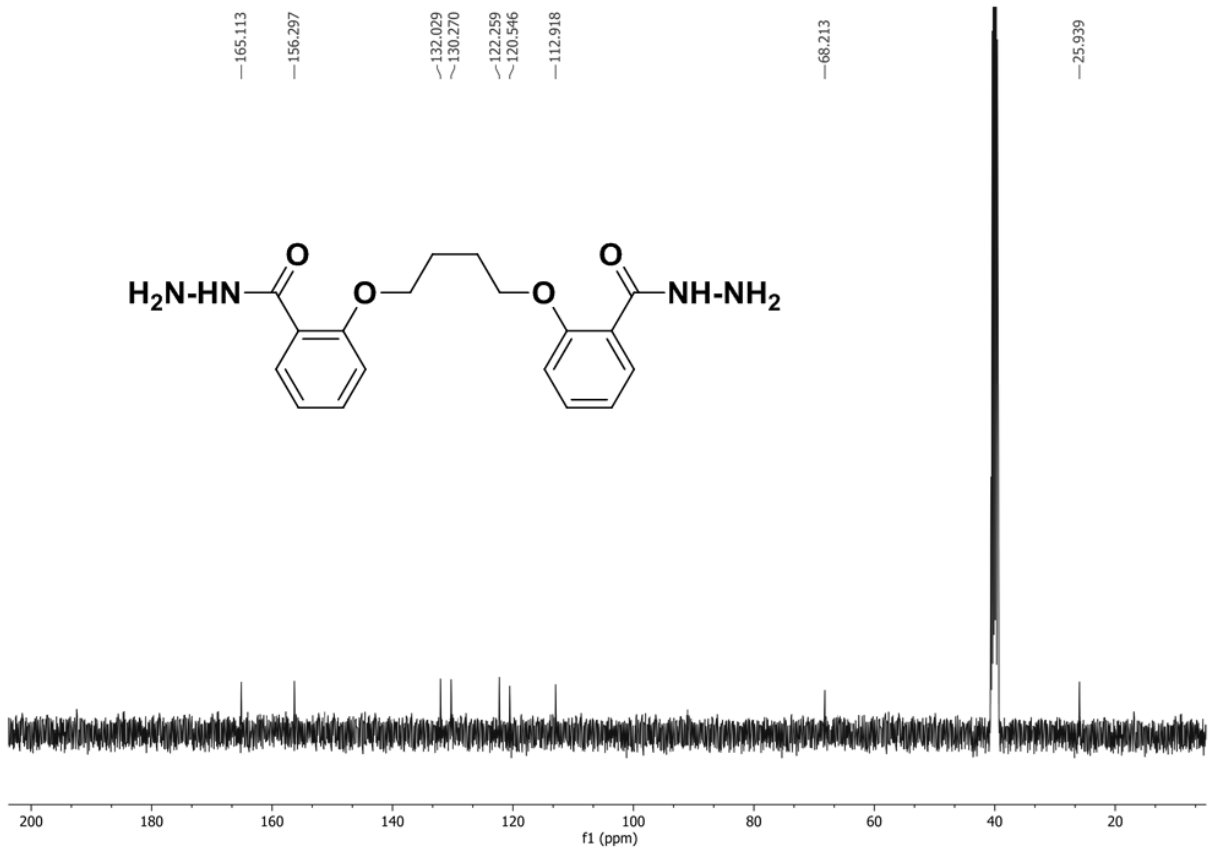
Synthesis of BDOBH.

A mixture of methyl salicylate (10 mmol) and 1,4-dibromobutane (5 mmol) in *N*,*N*-dimethylformamide (15 mL) in the presence of anhydrous potassium carbonate (15 mmol) was stirred at room temperature for six h. After adding 50 mL of water to the reaction mixture, 3 x 25 mL of chloroform were used to extract the mixture. The chloroform was evaporated under low pressure after the organic layers were combined and dried over sodium sulfate. After that, ethanol (15 mL) was used to re-dissolve the resulting crude bis(salicylates), and 4 mL of hydrazine hydrate was added. After that, the prior mixture was heated for 3 h at reflux. Filtration was used to gather the 2,2'-(butane-1,4-diylbis(oxy))di(benzohydrazide), which was then dried, washed with cold ethanol, and recrystallized using an equal volume of *N*,*N*-dimethylformamide and ethanol as colorless solid (87%); m.p. 305 ^{o}C; IR (υ cm^{-1}): 3230, 3072 (NH, NH_2), 3037, 2933, 2881, 1650 (CO), 1602, 1556, 1486, 1454, 1365, 1295, 1247, 1193, 1149, 1080, 748; ^{1}H-NMR (DMSO-d_6): δ

1.94 (br s, 4H, 2 OCH_2CH_2), 4.16 (br s, 4H, 2 OCH_2CH_2), 4.53 (s, 4H, 2 NH_2), 7.02 (t, J = 7.6 Hz, 2H, Ar-H), 7.14 (d, J = 7.6 Hz, 2H, Ar-H), 7.44 (t, J = 7.6 Hz, 2H, Ar-H), 7.66 (d, J = 7.6 Hz, 2H, Ar-H), 9.14 (s, 2H, 2 NH); ^{13}C-NMR (DMSO-d_6): δ 25.9, 68.2, 112.9, 120.5, 122.2, 130.2, 132.0, 156.2, 165.1; Anal. calcd. for $C_{18}H_{22}N_4O_4$ (358.4): C, 60.32; H, 6.19; N, 15.63; found: C, 60.43; H, 6.00; N, 15.81%.



The ^{1}H-NMR spectrum of compound **1** (DMSO-d_6; 400 MHz).



The ^{13}C-NMR spectrum of compound **1** (DMSO-d_6; 100 MHz).

References

Mekky, A. E. M., Sanad, S. M. H., & Abdelfattah, A. M. (2022). Tandem synthesis, antibacterial evaluation and SwissADME prediction study of new bis(1,3,4-oxadiazoles) linked to arene units. *Mendeleev Communications*, *32*(5). https://doi.org/10.1016/j.mencom.2022.09.014