

Catalytic Synthesis of Glycol Dicarbonate from Glycol and Dimethyl Carbonate by transesterification

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1. General information

DMC, diglycol (DGL), triglycol (TGL), tetraglycol (TTGL), AlCl_3 , 4A molecular sieve, ZnO , $(\text{CH}_3\text{COO})_2\text{Mg}$, CH_3COONa , CH_3COOK , MgO , $\text{Ca}(\text{OH})_2$, CaO , K_2CO_3 , CH_3ONa were purchased in chengdu kelon reagent factory and used without further treatment. ^1H NMR and ^{13}C NMR spectra were recorded in CDCl_3 on a Bruker AV300 NMR spectrometer with TMS as internal standard.

2. The Fractionation device

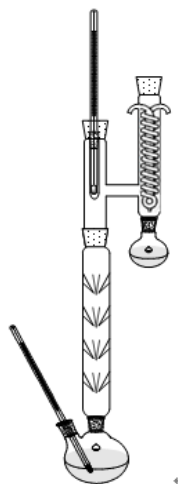


Fig.1 Fractionation device

3. The NMR spectra data of products

MC2: ^1H NMR(300 MHz, CDCl_3) δ (ppm): 4.22 (m, 2H), 3.70 (s, 3H), 3.64 (m,

4H), 3.51 (t, $J = 4.6$ Hz, 2H), 2.88 (d, $J = 4.7$ Hz, 1H). ^{13}C NMR(300 MHz, CDCl_3)

$\delta(\text{ppm})$: 155.6, 72.3, 68.6, 66.7, 61.3, 54.6.

DC2: ^1H NMR(300 MHz, CDCl_3) $\delta(\text{ppm})$: 4.27 (m, 4H), 3.77(s, 6H), 3.71(m, 4H).

^{13}C NMR(300 MHz, CDCl_3) $\delta(\text{ppm})$: 155.3, 68.4, 66.5, 55.3.

Dimer: ^1H NMR (300 MHz, CDCl_3) $\delta(\text{ppm})$: 4.24 (m, 8H), 3.74 (s, 6H), 3.66 (m, 8H). ^{13}C NMR (300 MHz, CDCl_3) $\delta(\text{ppm})$: 155.6, 154.9, 68.7, 66.8, 54.7.

DC3: ^1H NMR(300 MHz, CDCl_3) $\delta(\text{ppm})$: 4.12 (m, 4H), 3.62(s, 6H), 3.56 (m, 4H), 3.5 (s, 4H). ^{13}C NMR(300 MHz, CDCl_3) $\delta(\text{ppm})$: 155.3, 70.2, 68.5, 66.6, 54.3.

DC4: ^1H NMR(300 MHz, CDCl_3) $\delta(\text{ppm})$: 4.25 (m, 4H), 3.74(s, 6H), 3.68(m, 4H), 3.61(s, 8H). ^{13}C NMR(300 MHz, CDCl_3) $\delta(\text{ppm})$: 155.6, 70.5, 68.8, 66.9, 54.7.

4. The NMR spectrums of products

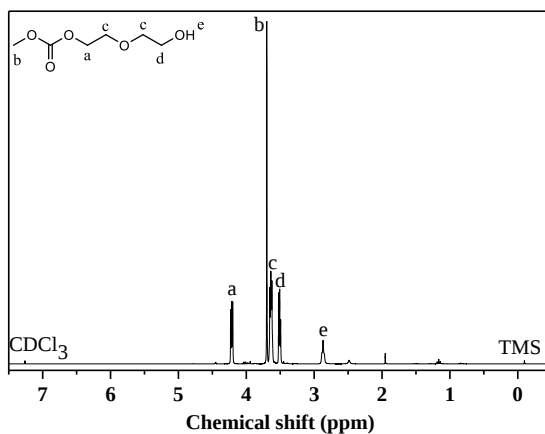


Fig.2 ^1H NMR of MC2

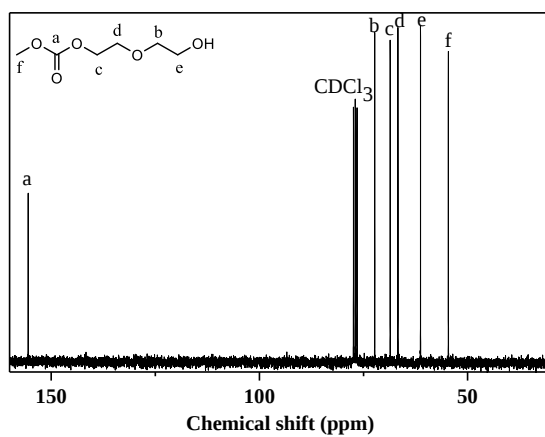


Fig.3 ^{13}C NMR of MC2

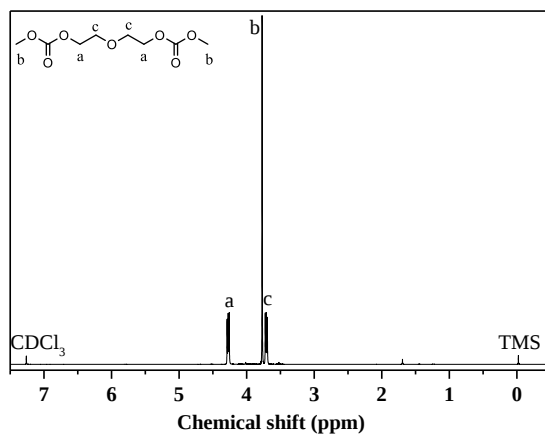


Fig.4 ^1H NMR of DC2

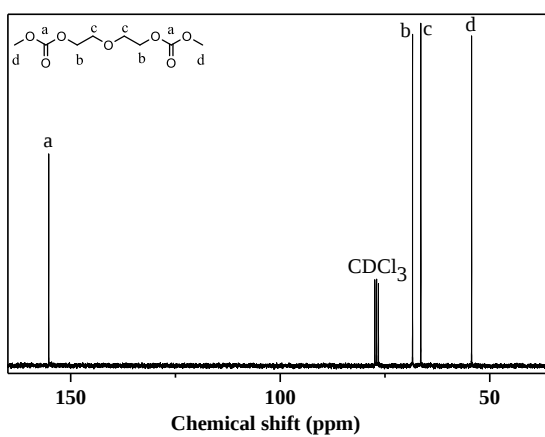


Fig.5 ^{13}C NMR of DC2

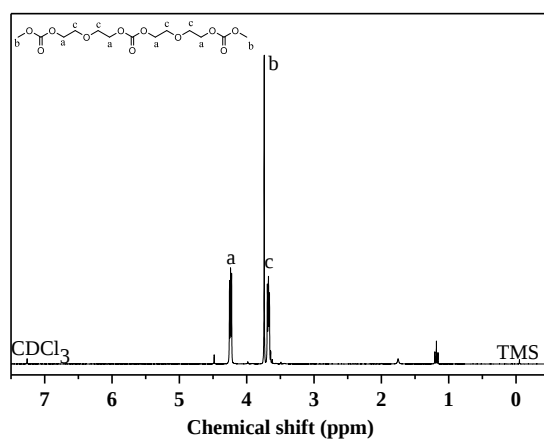


Fig.6 ¹H NMR of Dimer

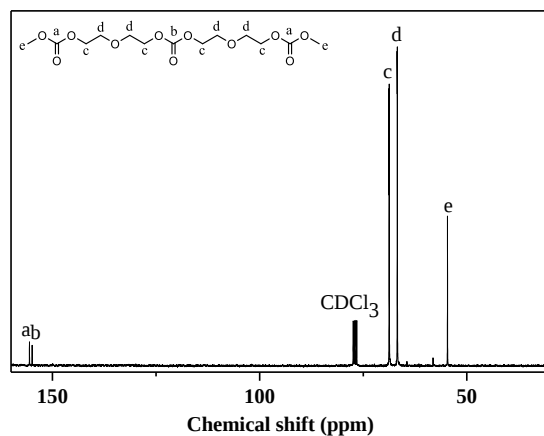


Fig.7 ¹³C NMR of Dimer

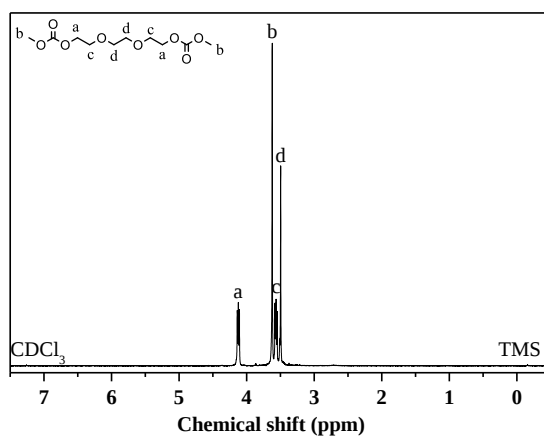


Fig.8 ¹H NMR of DC3

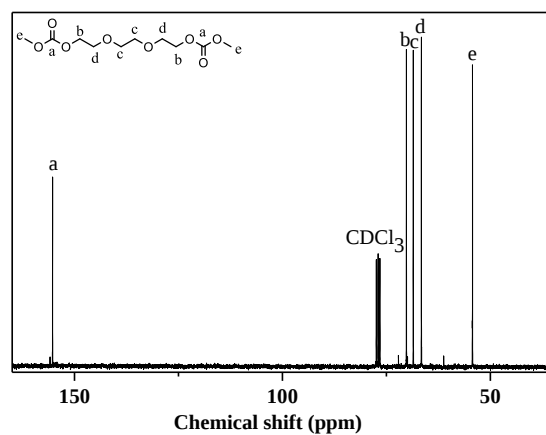


Fig.9 ^{13}C NMR of DC3

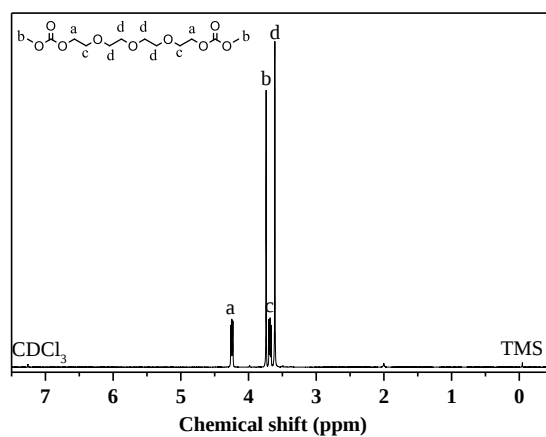


Fig.10 ^1H NMR of DC4

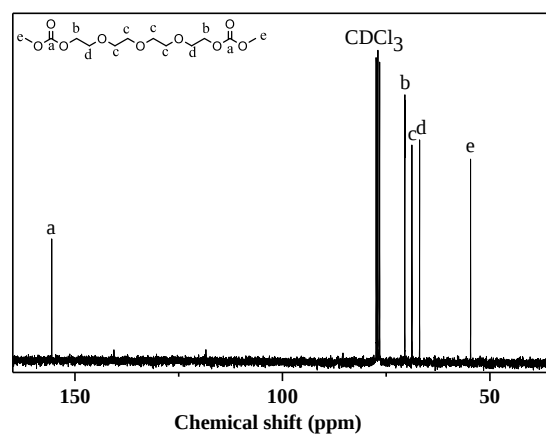


Fig.11 ^{13}C -NMR of DC4