CO_2 coupling with epoxides catalysed by using one-pot synthesised, *in situ* activated zinc ascorbate under ambient conditions

Feda'a M. Al-Qaisi,[*a] Abdussalam K. Qaroush,[*b] Amneh H. Smadi,[a] Fatima Alsoubani,[a] Khaleel I. Assaf,[c] Timo Repo[d] and Ala'a F. Eftaiha[*a]

[a] Department of Chemistry, The Hashemite University, P.O. Box 150459, Zarqa 13115, Jordan.

E-mails: fedaam@hu.edu.jo; alaa.eftaiha@hu.edu.jo

[b] Department of Chemistry, Faculty of Science, The University of Jordan, Amman 11942, Jordan.

E-mail: a.qaroush@ju.edu.jo

[c] Department of Chemistry, Faculty of Science, Al-Balqa Applied University, 19117 Al-Salt, Jordan.

[d] Department of Chemistry, University of Helsinki A.I. Virtasen aukio 1, 00014 Helsinki, Finland.

Electronic Supplementary Information (ESI)

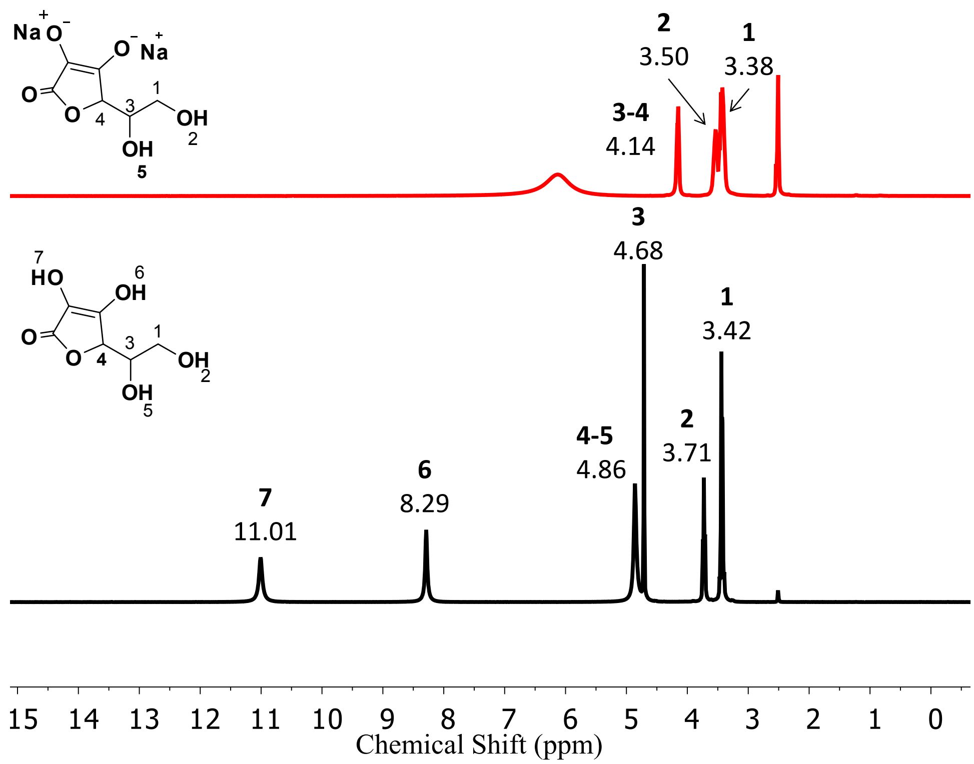

Fig. S1. [1]H NMR spectra (in DMSO-d_6) of $ASC(OH)_2$ (**black trace**) and $ASC(ONa)_2$ (**red trace**).

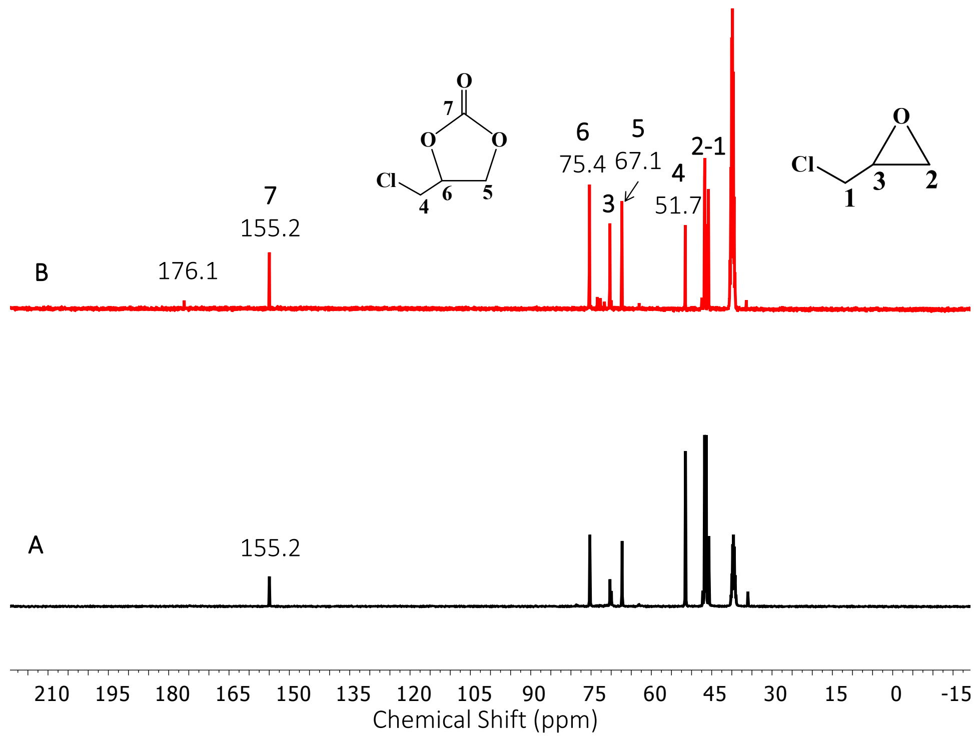

Fig. S2. ^{13}C NMR (in DMSO-d_6) spectra of ECH conversion after 17 h (A) and 41 h (B) reaction time, respectively.

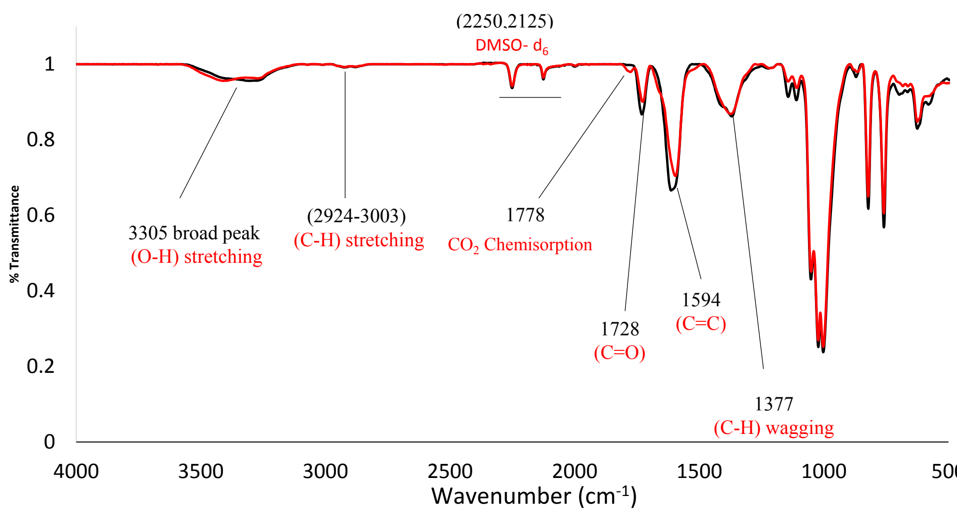

Fig. S3. ATR-FTIR spectra of ASC(O)$_2$Zn before (**black**) and after bubbling (**red trace**) CO_2.

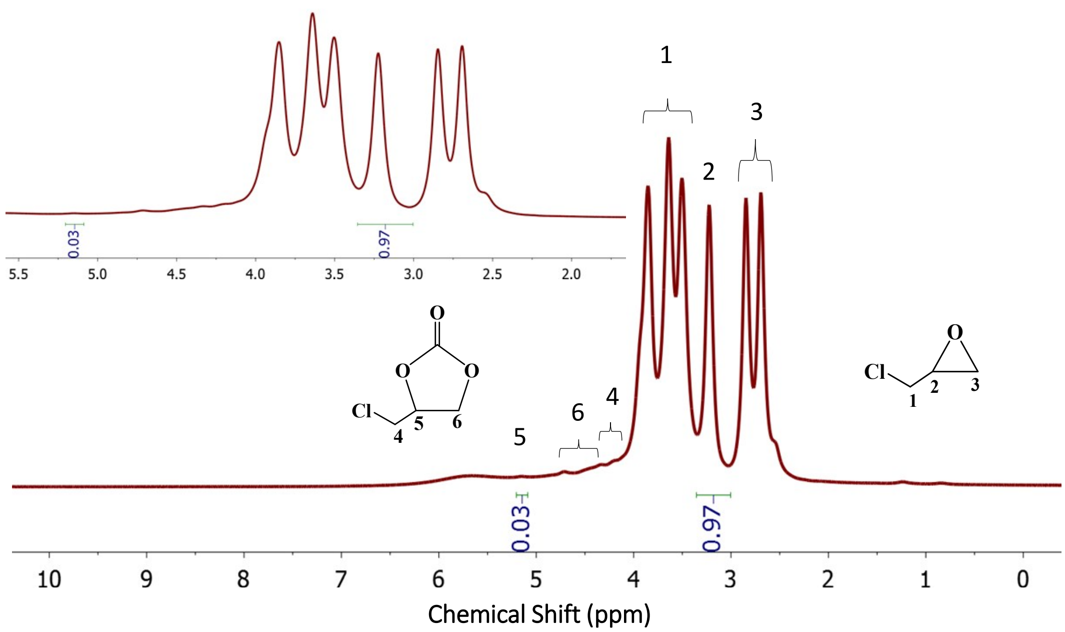

Fig. S4. ^{1}H NMR spectrum (in DMSO-d_6) of epichlorohydrin carbonate activated by $ZnCl_2$/sodium ascorbate (Reaction Conditions: 17 h and 25 ºC)

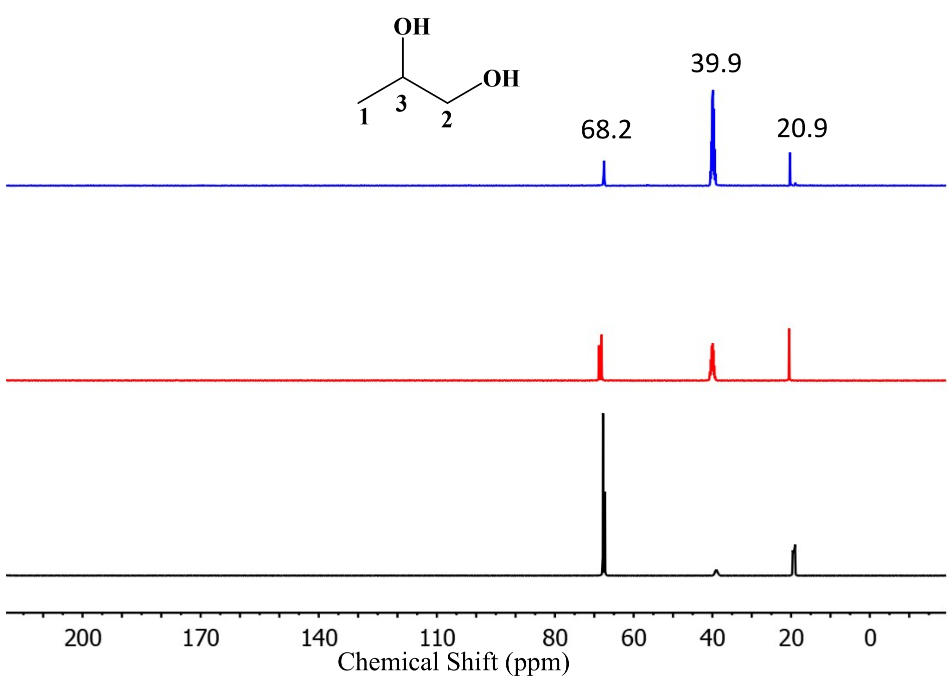

Fig. S5. ^{13}C NMR spectra (in DMSO-d_6) of PG (**black trace**), PG/NaH/CO_2 (**red trace**) and PG/NaH/CO_2/$ZnBr_2$ (**blue trace**)

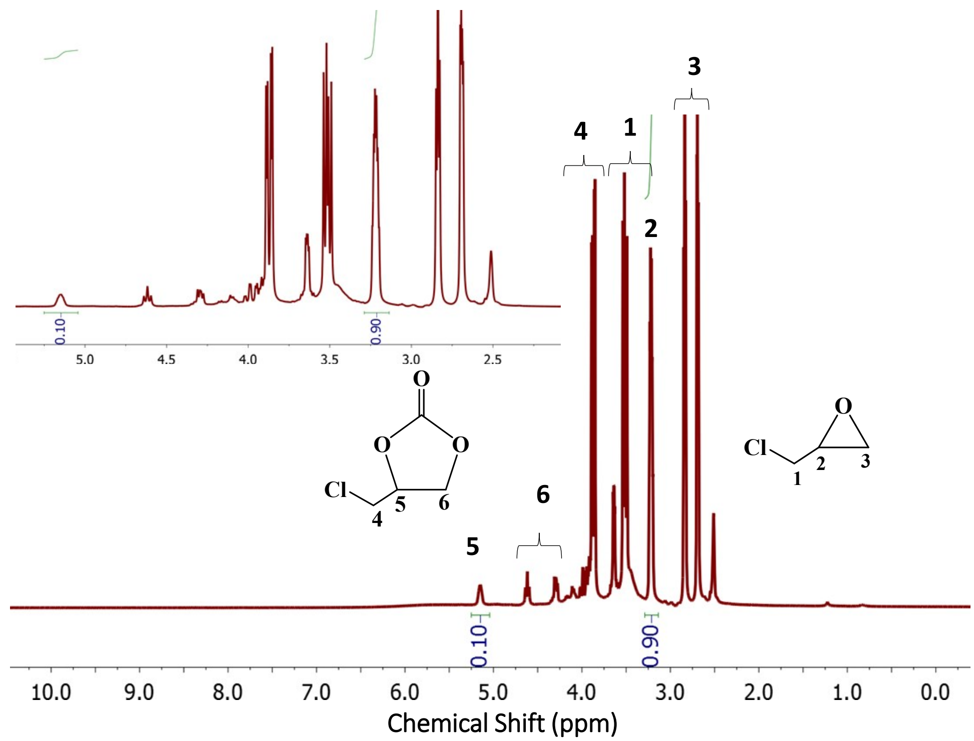

Fig. S6. ^{1}H NMR spectrum (in DMSO-d_6) of epichloro carbonate (ECC) entry 1. (Reaction Conditions: 17 h and 20 ºC).[1]

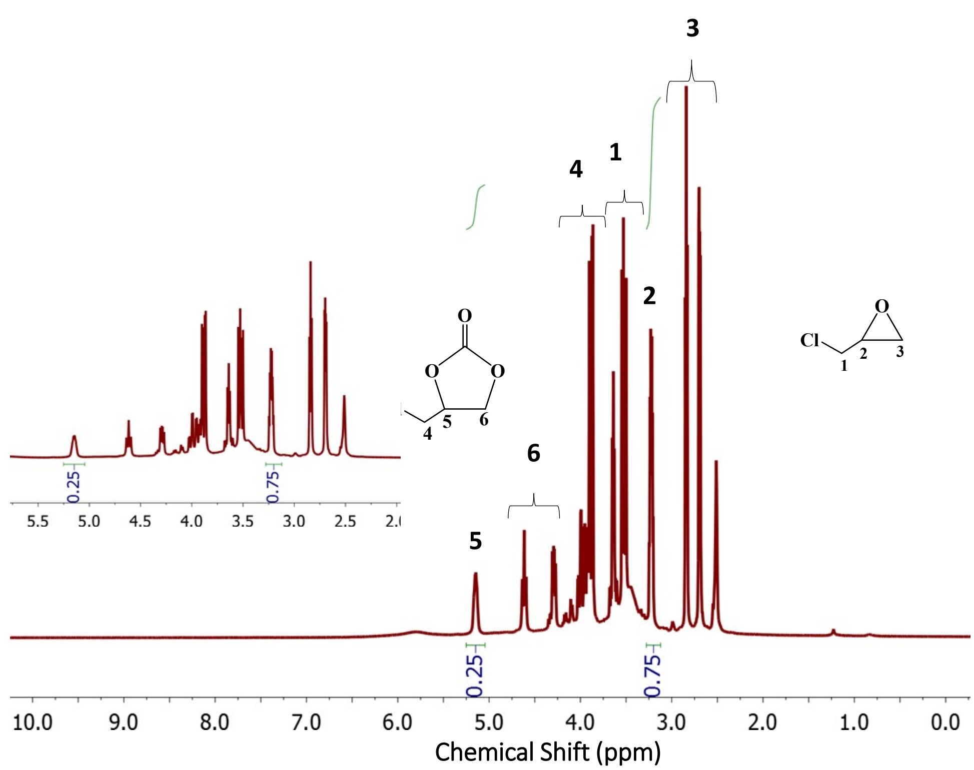

Fig. S7. ^{1}H NMR spectrum (in DMSO-d_6) of ECC entry 2. (Reaction Conditions: 17 h and 20 ºC).[1]

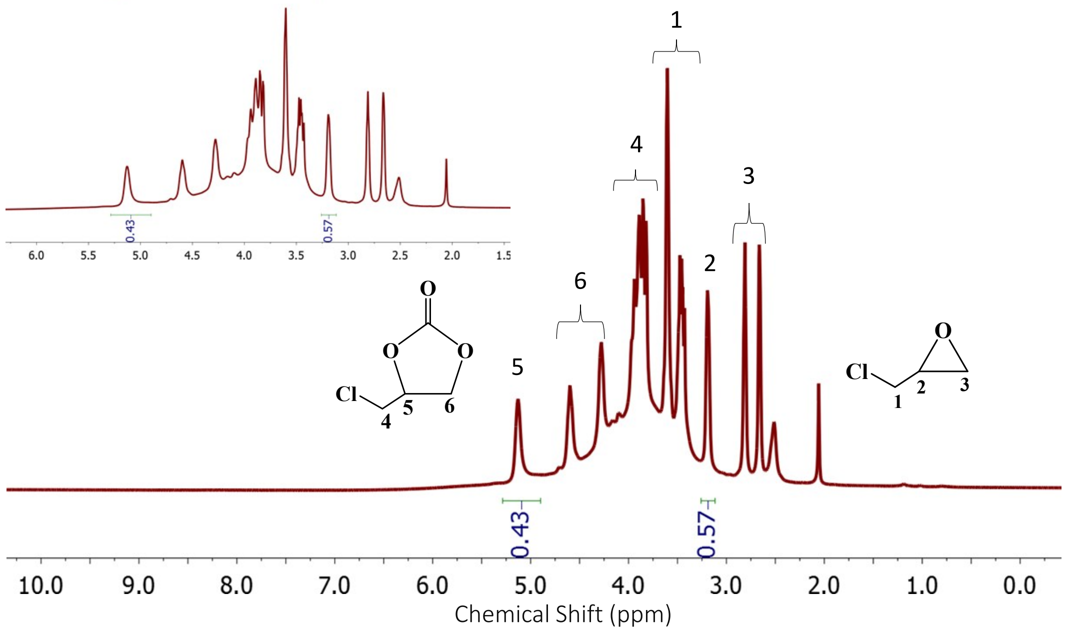

Fig. S8. ^{1}H NMR spectrum (in DMSO-d_6) of ECC entry 3. (Reaction Conditions: 17 h and 20 ºC). [1]

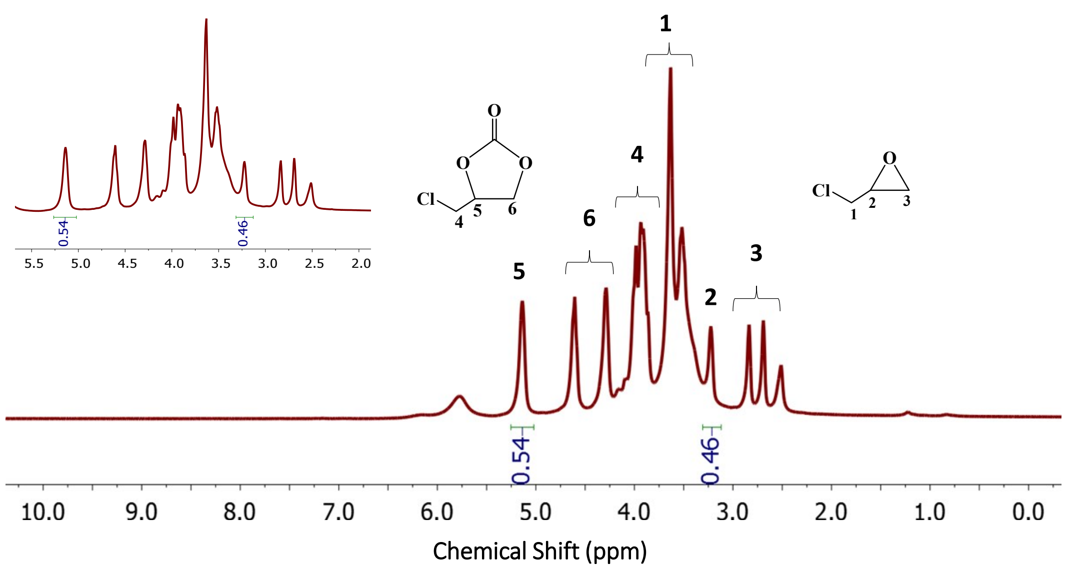

Fig. S9. ^{1}H NMR spectrum (in DMSO-d_6) of ECC entry 4. (Reaction Conditions: 41 h and 20 ºC). [1]

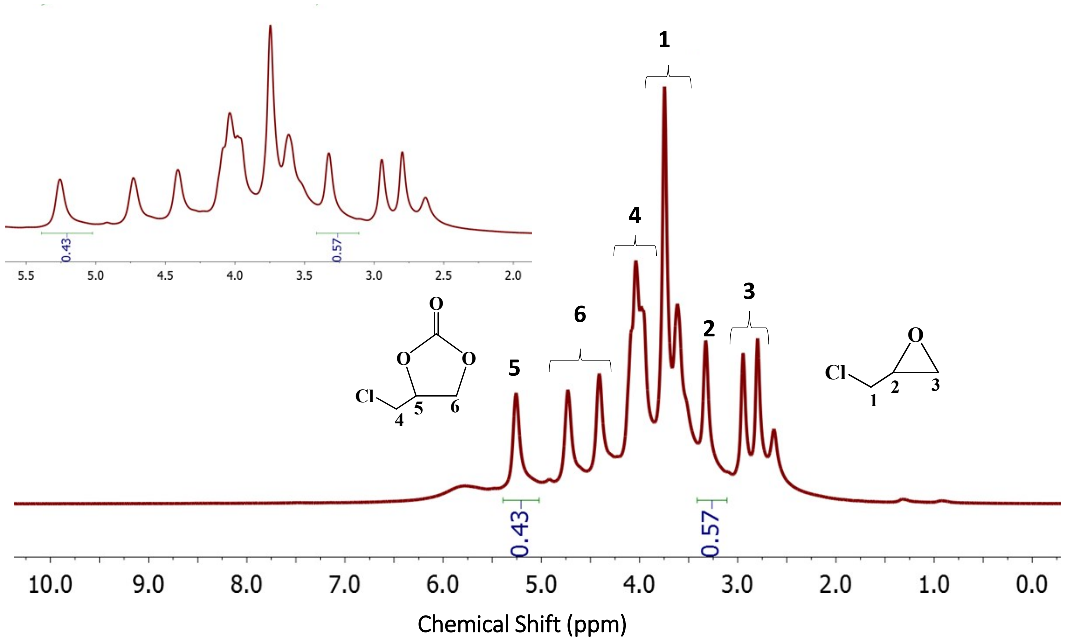

Fig. S10. ^{1}H NMR spectrum (in DMSO-d_6) of ECC entry 5 (Reaction Conditions: 17 h and 30 ºC).[1]

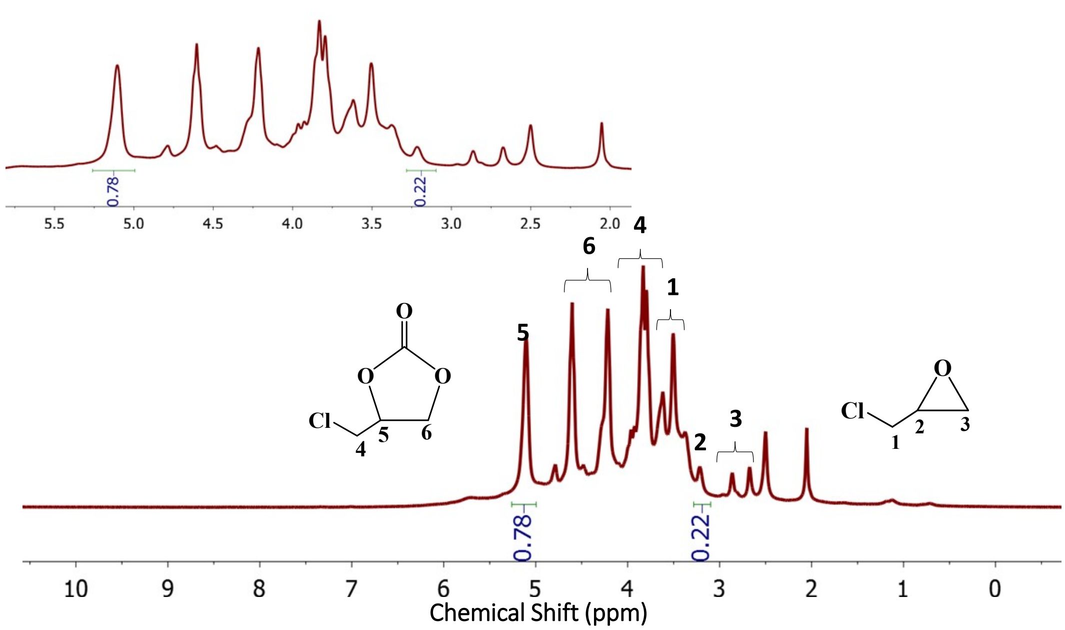

Fig. S11 . ^{1}H NMR spectrum (in DMSO-d_6) of ECC entry 6 (Reaction Conditions: 17 h and 50 ºC).[1]

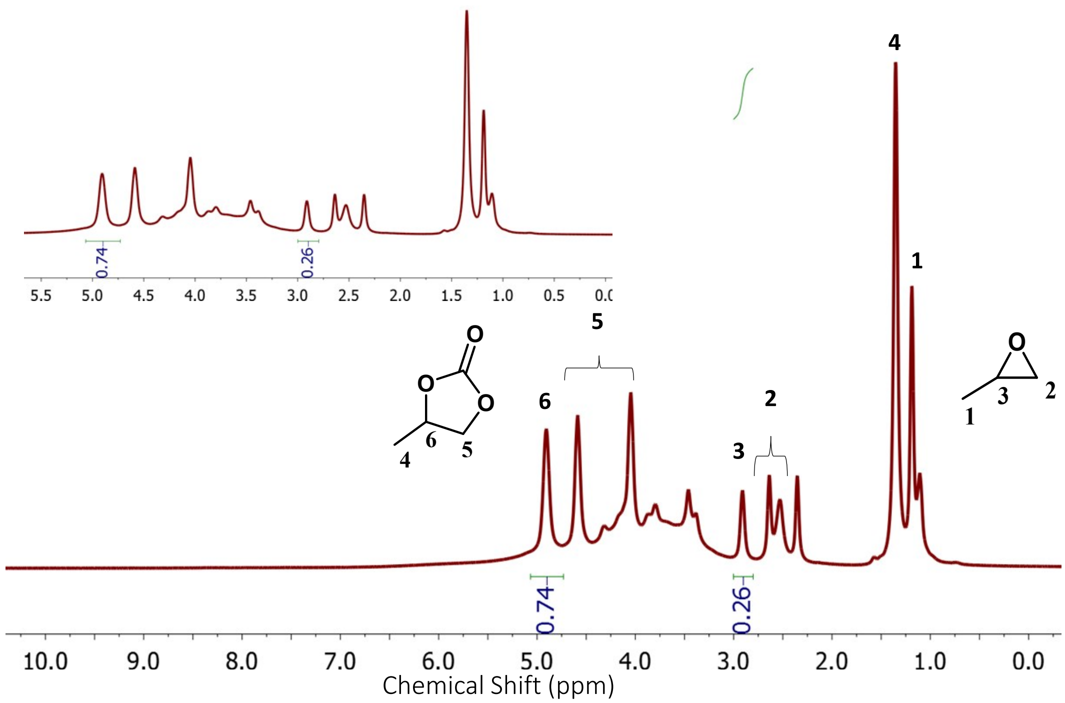

Fig. S12 . [1]H NMR spectrum (in DMSO-d_6) of propylene carbonate entry 7 (Reaction Conditions: 17 h and 20 ºC).[1]

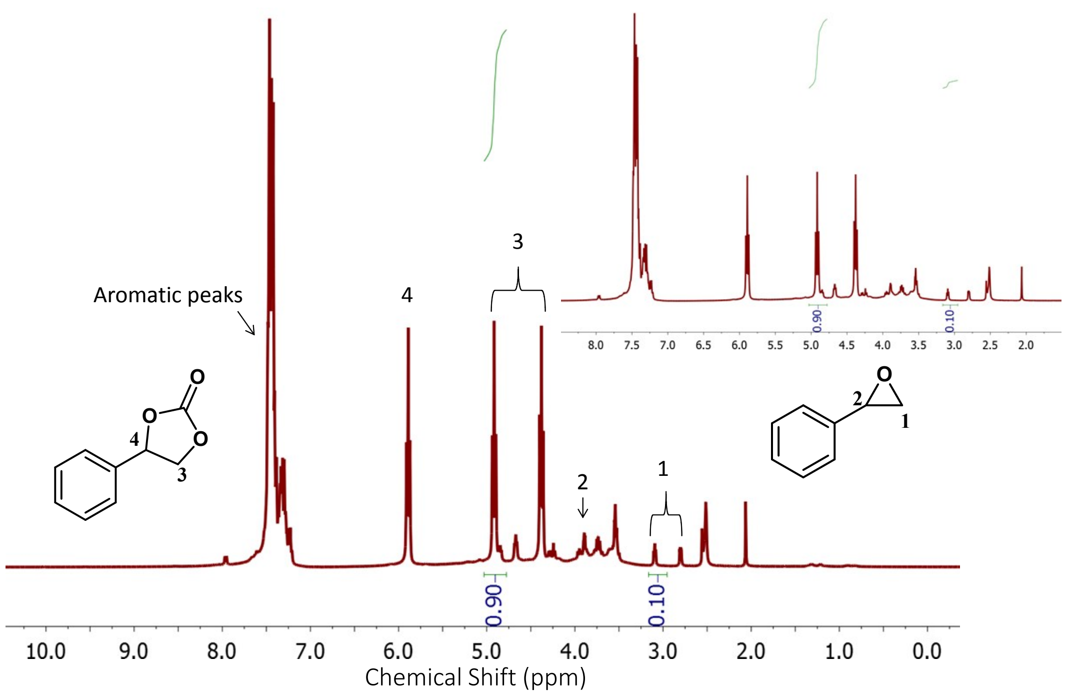

Fig. S13 . [1]H NMR spectrum (in DMSO-d_6) of styrene carbonate entry 8 (Reaction Conditions: 17 h and 50 ºC).[1]

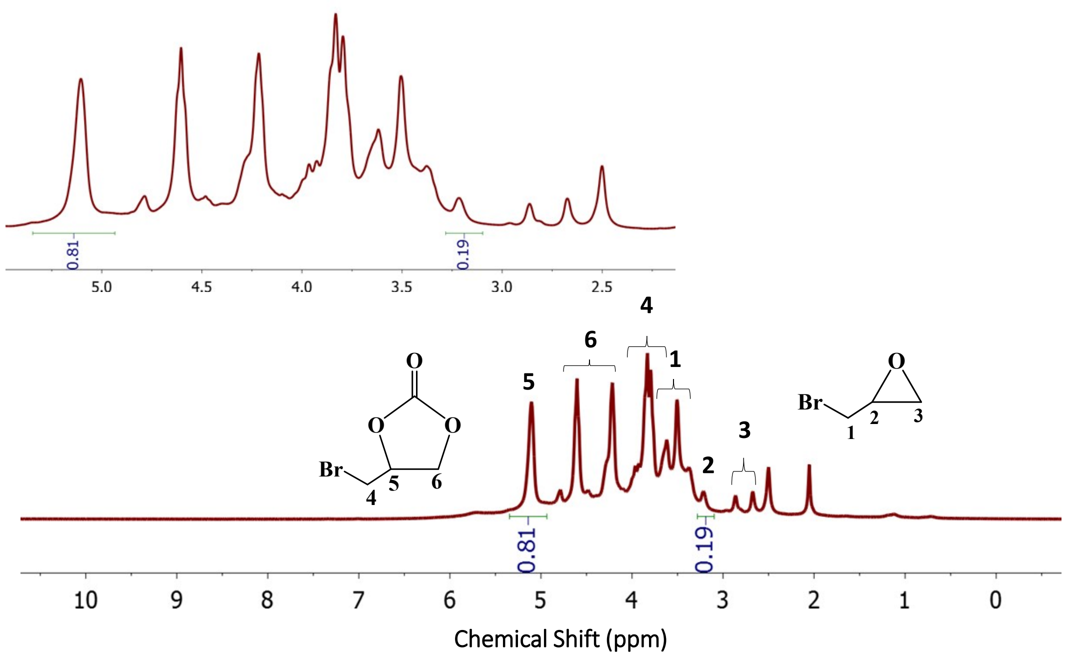

Fig. S14. ^{1}H NMR spectrum (in DMSO-d_6) of epibromo carbonate entry 9 (Reaction Conditions: 17 h and 50 ºC).[2]

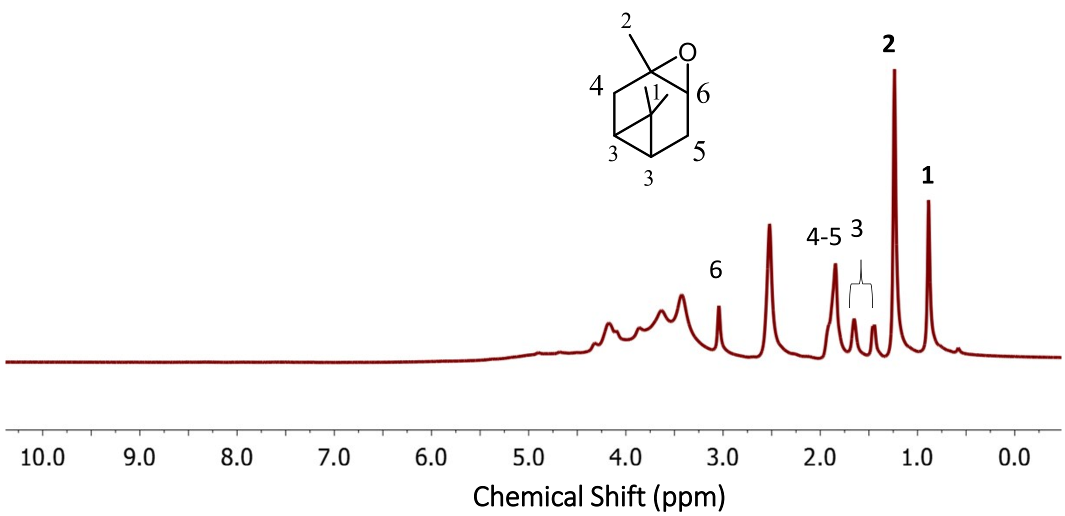

Figure S15 . ^{1}H NMR spectrum (in DMSO-d_6) of pinene oxide entry 10 (Reaction Conditions: 17 h and 50 ºC). Note: No signals above 5 in ^{1}H NMR for carbonate product**.** [3]

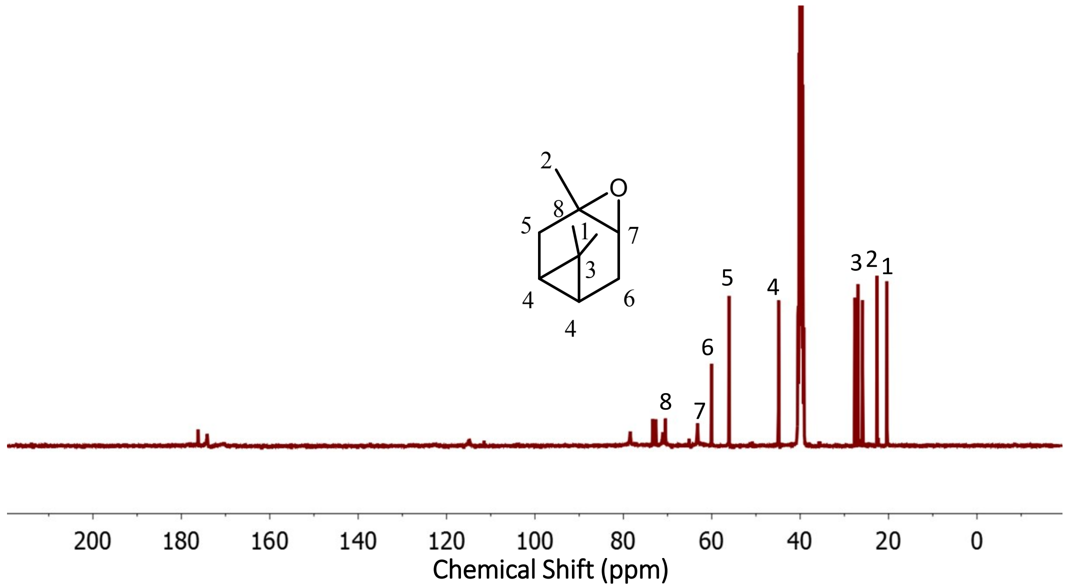

Fig. S16. ^{13}C NMR spectrum (in DMSO-d_6) of pinene oxide entry 10 (Reaction Conditions: 17 h and 50 ºC). Note: no appearance for signal in region between 150-160 ppm in ^{13}C NMR.[3]

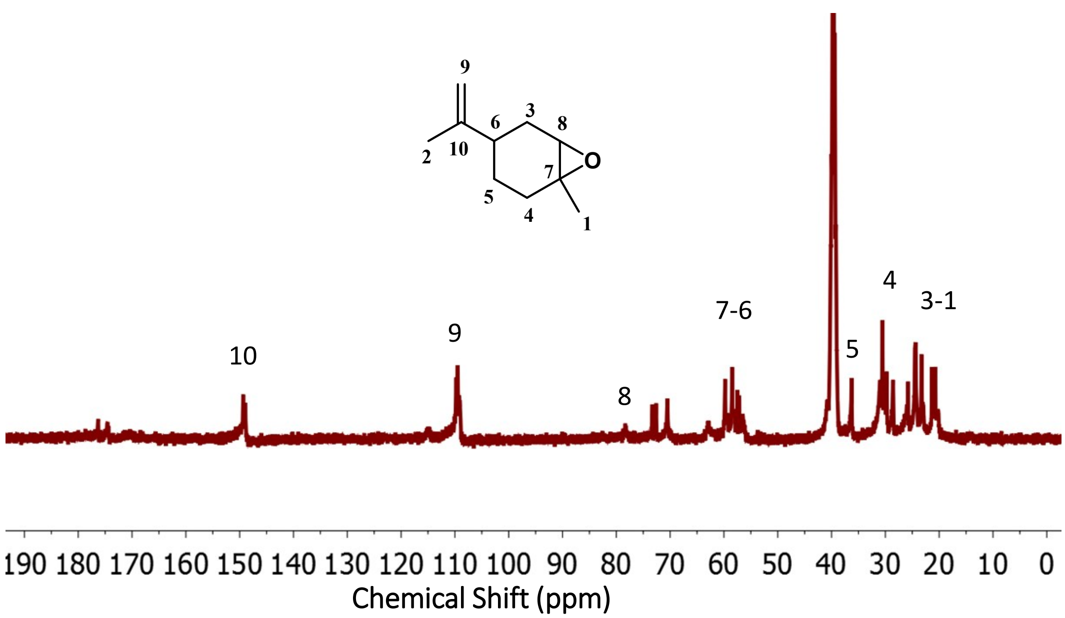

Fig. S17. ^{13}C NMR spectrum (in DMSO-d_6) of limonene oxide entry 11 (Reaction Conditions: 17 h and 50 ºC). Note: No product as no signals above 5 in ^{1}H NMR for carbonate product.[2]

(1) Zhang, S.; Han, F.; Yan, S.; He, M.; Miao, C.; He, L.-N. Efficient Catalysts In Situ Generated from Zinc, Amide and Benzyl Bromide for Epoxide/CO_2 Coupling Reaction at Atmospheric Pressure: Efficient Catalysts In Situ Generated from Zinc, Amide and Benzyl Bromide for Epoxide/CO_2 Coupling Reaction at Atmospheric Pressure. *Eur. J. Org. Chem.* **2019**, *2019* (6), 1311–1316. https://doi.org/10.1002/ejoc.201801653.

(2) Qaroush, A. K.; Alsoubani, F. A.; Al-Khateeb, A. M.; Nabih, E.; Al-Ramahi, E.; Khanfar, M. F.; Assaf, K. I.; Eftaiha, A. F. An Efficient Atom-Economical Chemoselective CO_2 Cycloaddition Using Lanthanum Oxide/Tetrabutyl Ammonium Bromide. *Sustainable Energy Fuels* **2018**, *2* (6), 1342–1349. https://doi.org/10.1039/C8SE00092A.

(3) Zhi, Y.; Shan, X.; Shan, S.; Jia, Q.; Ni, Y.; Zeng, G. Synthesis and Thermal Degradation Kinetics of New Terpolymer of Carbon Dioxide, Cyclohexene Oxide and Alpha-Pinene Oxide. *Journal of CO2 Utilization* **2017**, *22*, 299–306. https://doi.org/10.1016/j.jcou.2017.09.020.