Supporting Information

C_{60} fullerenol as highly efficient catalyst for synthesis of cyclic carbonates from CO_2 and epoxides

Yong-Bin Sun, Chang-Yan Cao*, Shu-Liang Yang, Pei-Pei Huang, Chun-Ru Wang and Wei-Guo Song*

Beijing National Laboratory for Molecular Sciences (BNLMS), Laboratory for Molecular Nanostructures and Nanotechnology, Institute of Chemistry, Chinese Academy of Sciences, Beijing 100190, P. R. China.

E-mail: cycao@iccas.ac.cn, wsong@iccas.ac.cn

1. Experimental section

Materials: CO_2 was purchased from Beijing Beiwen Gas Factory (≥99.5%). Toluene, ethanol, hydrogen peroxide (30 wt. %), MnO_2, diethyl ether, isopropanol, propylene oxide were purchased from Beijing Chemical Reagents Company. C_{60} fullerene was purchased from Suzhou Dade Carbon Nanotechnology Co., Ltd. Ozone was produced by Ozone Generator system. Epichlorohydrin, butyl glycidyl ether, 1,2-epoxyoctane, styrene oxide, 2-(prop-2-enoxymethyl)oxirane, and cyclohexene oxide were purchased from Alfa Aesar. Glycidyl phenyl ether was purchased from TCI (Shanghai) Chemical Industry Development Co., Ltd. All chemicals were used without further purification.

Preparation of C_{60} fullerenol: 0.2 g C_{60} fullerene was dissolved in toluene, and oxidized by ozone for 15 min under vigorous agitation. After the toluene was removed by rotary evaporation, the residual brown solids were dispersed by 50 mL ethanol, then added 40 mL hydrogen peroxide (30 wt.%). The mixture was vigorously stirred at 60 ^{o}C for 20 h, and it turned to be a deep red solution, then added 0.3 g MnO_2 under vigorous agitation for 4 h to accelerate hydrogen peroxide to decompose, at the same time, the solution became turbid. The precipitate was collected by centrifugation, and then added water to get the fullerenol aqueous solution. The fullerenol was precipitated by adding a mixed solvent of 2-propanol and diethyl ether (1:1). After centrifugation, the residual solid was washed twice with the mixed solvent of 2-propanol and diethyl ether (1:1), and then dried under vacuum at room temperature.

Cycloaddition reactions: All cycloaddition reactions were run in a 100 mL stainless-steel reactor equipped with a magnetic stirrer and automatic temperature control system (Parr 4560 with a Parr 4848 reactor controller, Parr Instrument Co.). An appropriate amount of CO_2 (2.0 MPa) was added into the autoclave preloaded with a mixture of epoxides (100 mmol), C_{60} fullerenol (100 mg), and KI (1 mmol) at room temperature. Then the temperature was raised to 120 °C and maintained for a desired time. The reactor was then cooled to room temperature in a water bath, and the remaining CO_2 was released slowly. The products were analyzed by GC (GC-2010 SHIMADZU) and GC-MS (SHIMADZU, GCMS-QP2010s). To study the reusability of the catalyst, the catalyst was washed three times with PO to remove the product and ethyl acetate.

Determination of PC yield: Propylene oxide (PO) is volatile, we use the external standard method to test the cyclic carbonate (PC) yield. Prepare the ethyl acetate solution with different concentration of PC, take 0.2 μL to be analyzed by GC, draw a line between PC peak area and PC concentration. The line as the standard to evaluate the yield of PC, as shown in Fig. S1.

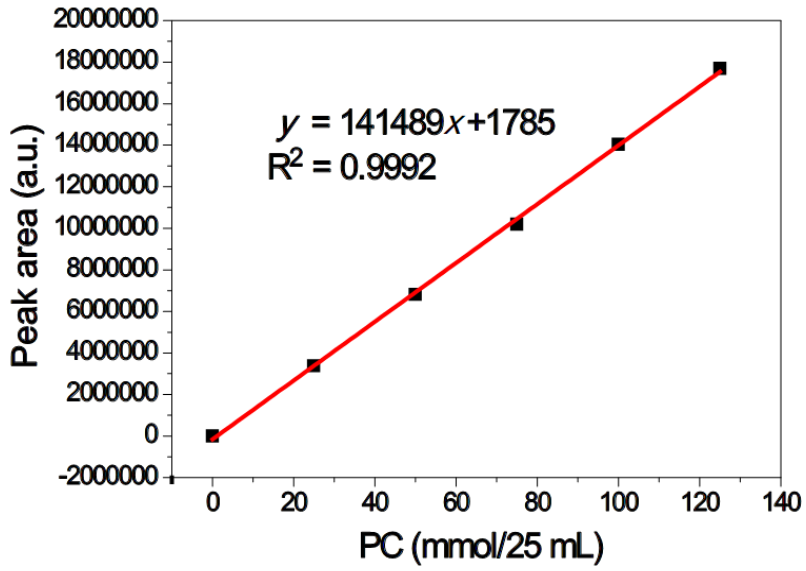

Fig. S1 The line between PC peak area and PC concentration.

2. Supporting figure and table

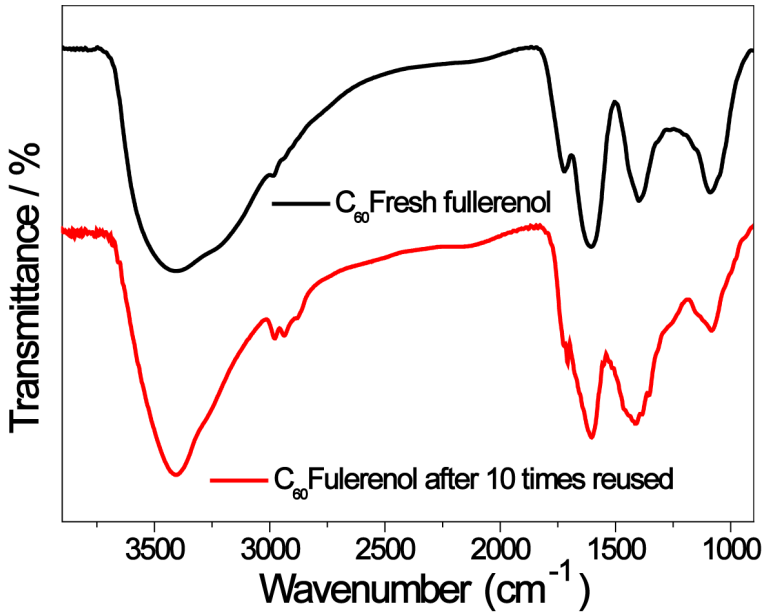

Fig. S2 IR spectrum of the fresh C_{60} fullerenol and C_{60} fullerenol after 10 times reused.

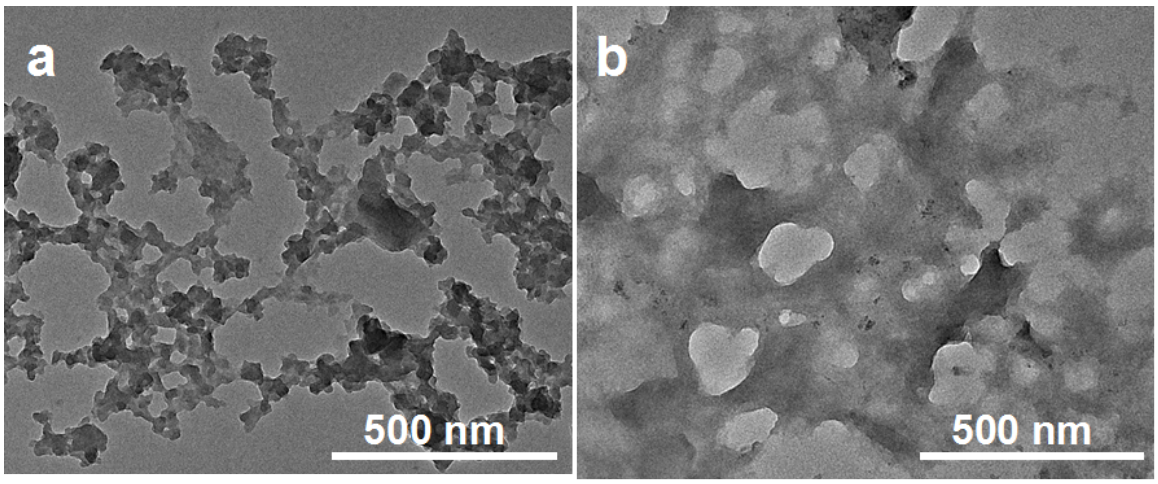

Fig. S3 TEM images of the fresh C_{60} fullerenol (a) and C_{60} fullerenol after 10 times reused (b).

Table S1 Elemental analysis of C_{60} fullerenol

Element	C	H	O
Percent / wt.%	45.2	4.0	50.8