

From Biomass to Functional Crystalline Diamond Nanothread: Pressure-Induced Polymerization of 2,5-Furandicarboxylic Acid

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Supporting Information

ABSTRACT: 2,5-Furandicarboxylic acid (FDCA) is one of the top-12 value-added chemicals from sugar. Besides the wide application in chemical industry, here we found that solid FDCA polymerized to form an atomic-scale ordered sp^3 -carbon nanothread (CNTh) upon compression. With the help of perfectly aligned π – π stacked molecules and strong intermolecular hydrogen bonds, crystalline poly-FDCA CNTh with uniform *syn*-configuration was obtained above 11 GPa, with the crystal structure determined by Rietveld refinement of the X-ray diffraction (XRD). The in situ XRD and theoretical simulation results show that the FDCA experienced continuous $[4 + 2]$ Diels–Alder reactions along the stacking direction at the threshold C...C distance of ~ 2.8 Å. Benefiting from the abundant carbonyl groups, the poly-FDCA shows a high specific capacity of 375 mAh g^{-1} as an anode material of a lithium battery with excellent Coulombic efficiency and rate performance. This is the first time a three-dimensional crystalline CNTh is obtained, and we demonstrated it is the hydrogen bonds that lead to the formation of the crystalline material with a unique configuration. It also provides a new method to move biomass compounds toward advanced functional carbon materials.

Production of green polymeric materials from biomass-based platform chemicals is necessary for a sustainable and greener society.^{1–4} 2,5-Furandicarboxylic acid (FDCA) is a well-known renewable building block that can be synthesized from oxidation of 5-hydroxymethylfurfural (HMF, an intermediate product of the acid dehydration of sugars) or directly from carbohydrates by a one-pot reaction.^{5,6} It has two carboxyl groups and can be transformed into several families of useful compounds, like the polyesters, polyurethanes, and polyamides.^{7–10} On the other hand, the furan ring is also an important precursor for constructing advanced materials. Very recently, furan was reported to transfer into sp^3 -carbon nanothreads (CNThs) via a pressure-induced polymerization (PIP) process under a relatively low pressure (~ 10 GPa) due to its reduced aromaticity, and a large-scale synthesis can thus be achieved.^{11,12} Such CNThs were also called diamond nanothreads,^{13–19} which were predicted to have extraordinary high tensile strength and bending modulus and have potential application in tunable thermal conductivity materials, energy storage devices, and nanoelectromechanical systems.^{20–22} Comparing to benzene,^{13–15} the furan ring has only four carbon atoms, resulting in fewer possible bonding routes and hence improving the homogeneity of the CNThs.²³ Under the help of the advanced solid-state nuclear magnetic resonance (NMR) techniques, Matsuura et al. concluded that about 10% of the sample is a perfect furan-CNTh, with an almost entirely *anti*-configuration.¹² Besides, the stacking order and intermolecular interaction (π – π stacking^{24–27} and H-bonding^{28–30}) of reactant molecules were also key factors in the preparation of structure-specific crystalline polymeric material, as evidenced by the PIP product of the naphthalene–

octafluoronaphthalene cocrystal,^{24,25} *s*-triazine,²⁶ pyridazine,²⁷ aniline,²⁸ acetylenedicarboxylic acid,²⁹ etc. Therefore, with the two pairs of intercarboxyl H-bonds, FDCA should be a better precursor for the synthesis of crystalline structure-specific CNThs. In this work, by compressing FDCA we obtained crystalline, carboxyl-substituted *syn*-furan-CNThs, as determined by the Rietveld refinement of X-ray diffraction (XRD) data, in obvious contrast to the furan-derived CNThs with *anti*-configuration.^{11,12} The carboxyl and the CNTh skeleton result in a good electrochemical performance and chemical stability for application as lithium-ion battery (LIB) anode material. Our work demonstrated a direct high-pressure synthetic route from biomass to advanced functional carbon-based material with excellent electrochemical properties and unique mechanical merit.

At ambient pressure, FDCA has a monoclinic lattice (Figure 1, space group $P2_1/m$) with $a = 4.97(1)$ Å, $b = 16.69(2)$ Å, $c = 3.66(1)$ Å, and $\beta = 96(1)^\circ$.³¹ The furan rings are stacked into columns with *syn*-configuration (all the oxygens on one side) along the *c*-axis, and these columns are connected by H-bonds along the *b*-axis. The angle between the furan plane and the *a*–*b* plane is 33.8° , and the minimum intermolecular distance between carbon atoms is $d_{C2...C3'} = 3.284$ Å. We investigated the high-pressure structural evolution of FDCA by in situ

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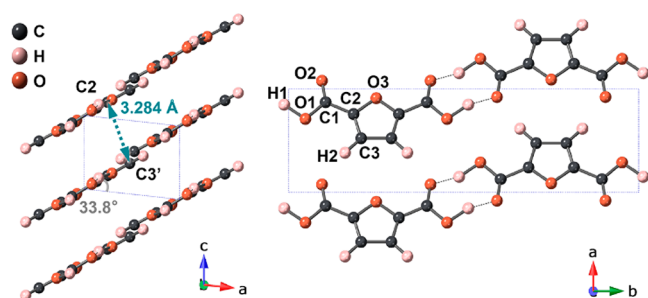


Figure 1. Crystal structure of FDCA at ambient pressure viewed along the *b*- and *c*-axis.

synchrotron XRD experiments (Figures 2a and S1). Upon compression, the diffraction peaks moved toward higher angles. It is worth noting that the peak 020 (index below the patterns in Figure 2a, at $\sim 4.3^\circ$) moved only a little bit, suggesting the structural stability of the *b*-axis (the direction of the H-bond). The 001 and -101 peaks moved the fastest, indicating the most pronounced deformation along the furan column. The cell parameters and volume of FDCA at high pressures were obtained by Le Bail fitting of the XRD data, which demonstrated the anisotropic compression quantitatively (Figure S2). At 12.1 GPa, new diffraction peaks appeared at 8.0° and 11.7° (marked by asterisks in Figure 2a), close to the peaks 110 and 140, respectively, and the sample became brown (Figure S3), which indicates the onset of reaction and the generation of poly-FDCA. These peaks were maintained at higher pressure and during decompression, and a series of diffraction peaks including $d = 8.45$, 5.18 , 4.96 , and 3.25 Å were clearly observed after decompression to 1.0 GPa.

The critical crystal structure of FDCA at 10.8 GPa (just before reaction, Figure 2b) was determined by Le Bail fitting

and subsequent density functional theory (DFT) optimization of the atomic coordinates. The FDCA molecule maintained in plane, and the $d_{C2\cdots C3'}$ between adjacent furan rings was compressed to 2.788 Å (green line in Figure 2b). This distance agrees with the reaction threshold ($d_{C\cdots C}$) of many aromatic molecules under high pressure, like $d_{C\cdots C} = 2.8$ Å for benzene³² and C_6H_6 – C_6F_6 cocrystals,³³ and hence suggests that FDCA can polymerize via a $[4 + 2]$ cycloaddition reaction by bonding of $C2\cdots C3'$, as confirmed by the solid-state NMR studies of the furan–CNTh.¹² Our dynamic simulation result also followed this route and produced the *syn*-FDCA–CNTh as expected (Figure S4).

To investigate the crystal structure of the poly-FDCA experimentally, we synthesized milligrams of the polymerized product by Paris–Edinburgh (PE) press at 30 GPa (noted as PE-30). Compared to the reported benzene/furan/thiophene-derived CNThs with only one broad diffraction peak at 4.9 – 5.6 Å,^{11,15,18} the poly-FDCA shows many sharp diffractions (Figure 2c and Figure S5 plotted in *d*-spacing) with a minimum *d*-spacing of ~ 1.4 Å distinguished. This unambiguously demonstrated the atomic-level ordering in the CNThs. Inspired by the theoretical simulation, a monoclinic unit cell was used to index the pattern perfectly, and the lattice parameters were obtained with $a = 5.3526(5)$ Å, $b = 16.9062(12)$ Å, $c = 2.6632(3)$ Å, $\alpha = \gamma = 90^\circ$, and $\beta = 90.23(9)^\circ$ by Rietveld refinement (Figures 2c and S5, Table S1). The intense peaks at 8.46 , 5.36 , and 2.66 Å are indexed as 020, 100, and 001, respectively, indicating that the crystal structure has excellent ordering in three dimensions (3D). The atomic coordinates were also determined by Rietveld refinement (Table S2), forming a *syn*-FDCA–CNTh structure. As shown in Figure 2d, the furan units form *syn*-CNThs along the *c*-axis and the H-bonds connect the CNThs in the *a*–*b* plane

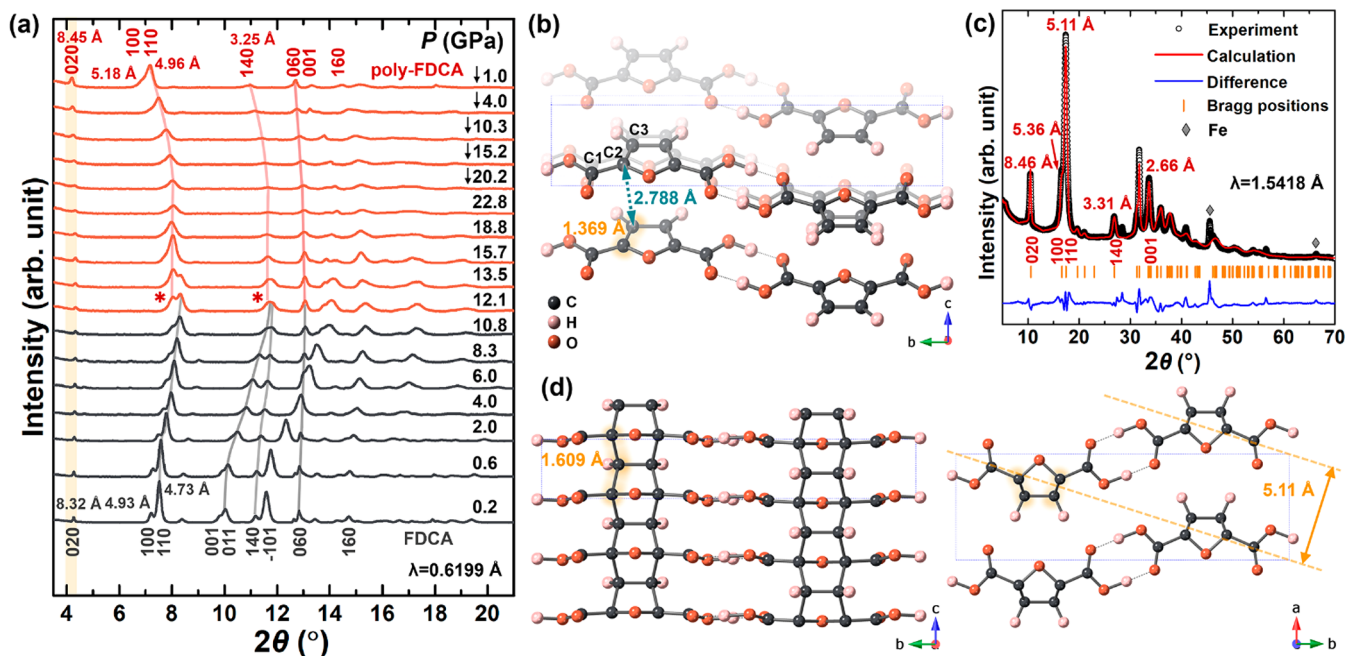


Figure 2. (a) Synchrotron XRD patterns of FDCA at selected pressures. The red asterisks indicate the formation of poly-FDCA, and down-arrows represent decompression. The gray and red lines represent the diffraction peak shifts of FDCA and poly-FDCA, respectively. The Miller indices and the corresponding *d*-spacings of FDCA and poly-FDCA are labeled. (b) The critical crystal structure of FDCA at 10.8 GPa. (c) Rietveld refinement plot of the XRD pattern. The diamond labels represent the diffraction peaks of iron chips from the gasket. (d) Crystal structure of *syn*-FDCA–CNTh.

to form a network as in the reactant, in agreement with the dynamic simulation results mentioned above. This is the first time the atomic-level structure of CNTh was determined due to its enhanced structural ordering. The poly-FDCA has a unique *syn*-configuration and complete interthread ordering, in obvious contrast to the furan-CNThs and other CNThs. This is attributed to the in-phase columnar stacking of the FDCA monomer, which is locked by the H-bonds between carboxyl groups and cannot rotate during the polymerization.

The polymerization process of FDCA was also tracked by the in situ infrared (IR) spectroscopy (Figures 3a and S6a and

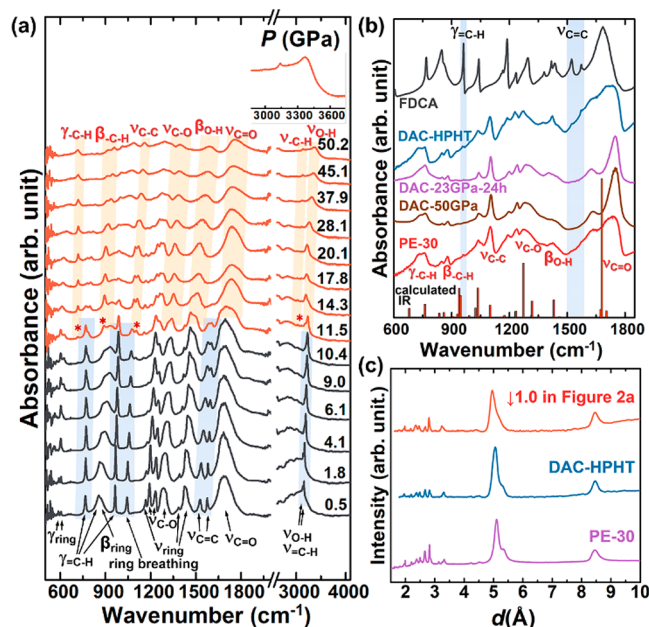


Figure 3. (a) In situ IR spectra of FDCA upon compression. The inset shows the amplified spectrum at 50.2 GPa; the blue and yellow regions mark the characteristic IR peaks of the FDCA and poly-FDCA, respectively. ν , γ , and β represent the stretching, out-of-plane bending, and in-plane bending vibration, respectively. (b) IR spectra collected at ambient pressure and room temperature for FDCA and poly-FDCA synthesized at various conditions, and the calculated IR spectrum of the *syn*-FDCA-CNTh. (c) XRD patterns of DAC-HPHT and PE-30 at ambient pressure and room temperature, with the in situ DAC sample decompressed to 1 GPa for comparison.

b) with the IR modes assigned in Table S3. Above 11.5 GPa, the characteristic IR modes of the furan ring including the out-of-plane bending of $\text{C}=\text{H}$ ($\gamma_{\text{C}=\text{H}}$, 771, 905, and 987 cm^{-1}) and the stretching vibration of $\text{C}=\text{C}$ ($\nu_{\text{C}=\text{C}}$, 1584 and 1603 cm^{-1}) gradually disappeared. Meanwhile, four new peaks appeared at 716, 889, 1103, and 3106 cm^{-1} , which are assigned to the $\text{C}-\text{H}$ out-of-plane bending mode ($\gamma_{\text{C}-\text{H}}$), $\text{C}-\text{H}$ in-plane bending mode ($\beta_{\text{C}-\text{H}}$), $\text{C}-\text{C}$ stretching mode ($\nu_{\text{C}-\text{C}}$), and $\text{sp}^3\text{-C}-\text{H}$ stretching mode ($\nu_{\text{C}-\text{H}}$), respectively (red asterisks in Figures 3a and S6b). Furthermore, the profiles of the ν -ring and $\text{C}=\text{O}$ stretching ($\nu_{\text{C}=\text{O}}$) modes at 1465 and 1696 cm^{-1} start to change significantly. All of these clearly confirmed the onset of the reaction of FDCA, with the transformation from sp^2 -carbon to sp^3 -carbon. The pressure dependence of selected IR peaks also shows obvious discontinuity or new peaks at 10–12 GPa, demonstrating the PIP clearly (Figure S6c). During decompression (Figure S6d) down to ambient pressure (DAC-50 GPa in Figure 3b), the vibration modes ($\gamma_{\text{C}-\text{H}}$, $\beta_{\text{C}-\text{H}}$, $\nu_{\text{C}-\text{C}}$, $\nu_{\text{C}-\text{H}}$) of poly-FDCA

were maintained, and the $\gamma_{\text{C}=\text{H}}$ and $\nu_{\text{C}=\text{C}}$ modes of FDCA did not reappear, indicating the irreversibility of the reaction and the stability of the CNTh. Moreover, the IR spectra of DAC-50 GPa and PE-30 (Figure 3b) are nearly identical, indicating that the polymers made from DAC and PE press have the same structure. The calculated IR spectrum of *syn*-FDCA-CNTh makes an excellent match to the experimental IR spectrum, which further validates the model (Figure 3b).

The synthetic pressure of poly-FDCA was significantly reduced by increasing the reaction time and temperature, as confirmed by the IR spectra and XRD (Figures 3b,c and S7). The samples obtained using DAC at 50 GPa (DAC-50 GPa), at 23 GPa for 24 h (DAC-23 GPa-24h), at 350 $^{\circ}\text{C}$ and 12 GPa (DAC-HPHT), respectively, and PE-30 have similar IR spectra, and the featured peak of FDCA like $\gamma_{\text{C}=\text{H}}$ and $\nu_{\text{C}=\text{C}}$ (marked in blue in Figure 3b) already disappeared. The XRD data of DAC-HPHT are consistent at different positions, indicating a good homogeneity (Figure S8). The patterns are almost identical to that of PE-30 and can be fitted with the *syn*-FDCA-CNTh model (Figures 3c and S9, Tables S4 and S5). It is worth noting that this is a milder condition for scalable preparation.

The abundant carbonyl groups bring redox properties to poly-FDCA, which provides a binding site for Li^+ ions through reversible electron transfer. We evaluated the electrochemical performance of PE-30 as an LIB anode material by fabricating half-cells. As shown in Figure 4a, the cyclic voltammogram

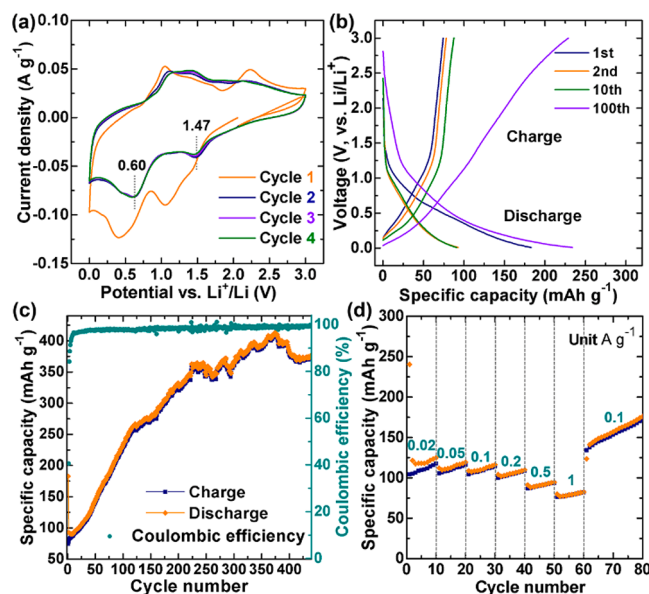


Figure 4. Electrochemical performance of PE-30 as an LIB anode material. (a) CV curves at a scan rate of 0.5 mV s^{-1} . (b) Galvanostatic discharge-charge profiles and (c) electrochemical cycling performance at a current density of 0.1 A g^{-1} . (d) Rate performance under different current densities (A g^{-1}).

(CV) curves show a pronounced reduction peak at 0.41 V in the first cycle, attributed to the formation of a solid electrolyte interface (SEI) film on the surface and the irreversible bonding of Li^+ ions to the carboxyl groups. This is consistent with the first charge-discharge profile shown in Figure 4b, corresponding to a Coulombic efficiency of 40.57%. In the following cycles, two cathodic peaks were steadily observed at ~ 0.60 and ~ 1.47 V owing to the reduction of carbonyl groups upon

insertion of Li^+ ions. The capacity and Coulombic efficiency increased dramatically upon cycling (Figure 4c), suggesting continuous activation of the poly-FDCA material and reversible insertion of Li^+ ions. The discharge/charge specific capacity reached a maximum of $\sim 413 \text{ mAh g}^{-1}$ at the 374th cycle and maintained $\sim 375 \text{ mAh g}^{-1}$ with a Coulombic efficiency above 99% in the subsequent cycles. This is close to the capacity of graphite (specific capacity of $\sim 360 \text{ mAh g}^{-1}$ by the formation of LiC_6),³⁴ which is the most commonly used anode material for lithium ions today. By reducing the constant current to 0.1 A g^{-1} after charging and discharging at a high rate of 1 A g^{-1} (Figure 4d), the discharge specific capacity was recovered to 141.1 mAh g^{-1} , reflecting good cycle/rate performance and structural stability. These results demonstrated that the poly-FDCA-CNTh is a good candidate as an LIB anode material.

In summary, we synthesized crystalline carboxyl-substituted *syn*-furan-CNTh with uniform chemical structure and excellent electrochemical properties as an LIB anode. The product shows distinct XRD peaks in three dimensions, which allowed determining the atomic positions by Rietveld refinement. The continuous $[4 + 2]$ cycloaddition reactions along the furan columns were concluded based on the structural analysis under threshold pressure and IR spectral evolution. The H-bonds guided the polymerization pathway to obtain the structure-specific crystalline product. The chemical stability of the sp^3 -CNTh backbone and the high content of carbonyl groups resulted in high electrochemical specific capacity and excellent cycling stability and evidenced that poly-FDCA is a good candidate for the LIB anode materials. This is the first example to show the application of the functionalized diamond nanothreads. Due to their high stiffness, better water affinity, and enhanced intermolecular interaction, more applications like reinforcement for the nanocomposites and development of biocompatible materials can be expected. More importantly, since H-bonds are very common in biomass molecules, and the molecules are often unsaturated, ready for polymerization upon compression, our work actually provides a high-pressure route for tailored synthesis of functional carbon-based materials from biomass.

■ ASSOCIATED CONTENT

SI Supporting Information

The Supporting Information is available free of charge at <https://pubs.acs.org/doi/10.1021/jacs.2c08914>.

Experimental procedures; synchrotron XRD patterns of FDCA under applied pressures; evolution of corresponding compression ratio of lattice parameters; P – V relationship of FDCA fitted by the third-order Birch–Murnaghan equation of state; optical micrographs of FDCA powder collected at selected pressures; poly-FDCA model obtained by dynamic simulation; XRD pattern of PE-30; IR investigation of FDCA under external pressure; IR spectra collected at ambient pressure and room temperature of the products synthesized at high pressure and room temperature and high pressure and high temperature; XRD data of poly-FDCA synthesized at 12 GPa and 350 °C; Rietveld refinement plot of the XRD pattern of product DAC-HPHT; Rietveld refinement results of PE-30 and DAC-HPHT based on the XRD data; atomic coordinates of

PE-30 and DAC-HPHT; assignments of IR modes of FDCA (PDF)

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Author Contributions

The manuscript was written through contributions of all authors. All authors have given approval to the final version of the manuscript.

Notes

The authors declare no competing financial interest.

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