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COMMUNICATION

A Metal-Free, High Nitrogen-Doped Nanoporous Graphitic Carbon Catalyst for an Effective Aerobic HMF-to-FDCA Conversion

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We report a metal-free catalysis of aerobic oxidation of 5-hydroxymethylfurfural (HMF) to 2,5-furandicarboxylic acids (FDCA), employing zeolitic-imidazole framework (ZIF-8) derived, nitrogen-doped nanoporous carbon (denoted as NNC) as the effective heterogeneous catalyst. The effect of high graphitic nitrogen loading in the NNC on the catalytic production of FDCA was demonstrated and discussed.

The sustainable production of transportation fuels and value added chemicals from renewable biomass resources has attracted great attention due to diminishing fossil resources.[1-3] Highly valued chemical components such as 5-hydroxymethylfurfural (HMF) and its derivative furan compounds (i.e. 2,5-dimethylfuran (DMF), 2,5-furandicarboxylic acid (FDCA), and γ-valerolactone (GVL)) converted from lignocellulosic biomass, have been demonstrated through various catalytic processes.[4-9] Among these biochemicals, 2,5-furandicarboxylic acid (FDCA) has been regarded as an important bio-based monomer for synthesizing valuable polymers,[10-14] especially polyethylene furanoate (PEF), which is considered to be a promising alternative to the most abundantly used polymer, polyethylene terephthalate (PET), for bottle production.

FDCA has been commonly oxidized from HMF by using diverse catalysts, including stoichiometric oxidants,[15] homogeneous metal salts,[16] enzyme,[17] and heterogeneous metal[18-21] or metal oxide catalysts.[22] Among them, noble metal catalysts (i.e. Pd, Au, Pt, and Ru) have been widely used as efficient catalysts in various catalytic systems.[23] However, the high costs of employing noble metal catalysts greatly inhibit development in industrial applications. Additionally, the leakage of metal ions from metal-based catalysis into reaction systems, could cause not only difficult purification of products but also serious environmental pollution.[23] For economic cost and green chemistry, the development of metal-free and effective heterogeneous catalyst for the aerobic oxidation of HMF to FDCA is extremely important.

Nitrogen-doped carbon materials (NCs) have attracted enormous interest in electrochemistry[24-26] and catalysis[27-29], owing to its easy preparation process, low cost, and outstanding catalytic performance. NCs are commonly synthesized through the post-treatment of previously prepared porous carbon with a nitrogen source (e.g. NH_3),[30] but this typically involve the drawbacks of lower nitrogen concentration and non-uniform nitrogen distribution. Recently, the synthesis of metal-organic frameworks (MOFs) inspires new approaches for making NCs. By using nitrogen (N)-containing organic ligands (i.e. imidazoles), zeolitic imidazole frameworks (ZIFs) with uniform and high N loading have been synthesized.[31] For example, ZIF-8 can be formed using zinc ions as metal nodes and 2-methylimidazole as organic ligands, and its preparation can be easily done in aqueous or organic solvent systems.[32, 33] Like other MOFs materials, ZIF-8 exhibits high specific surface area and well-defined morphology. In addition, after careful thermal treatments, one can easily remove metal ions and carbonize the framework, while retaining nitrogen in the framework to result in nitrogen-containing nanoporous carbon (NNC) nanoparticles.[34-36] Unlike conventional NC using post-treatment to introduce nitrogen into carbon framework, ZIF-8 derived NNC materials directly implement nitrogen during synthesis, thus enabling the encapsulation of nitrogen into carbon framework with high uniformity and quantity. We and others have previously applied functional NNC materials for catalytic reactions[37] and capacitive deionization of wastewater.[38] Since many nitrogen-doped carbon materials have so far been reported as efficient catalysts for selective oxidation of hydrocarbon such as benzylic alcohol,[39] ethylbenzene,[40] xanthene,[41] and Cumene,[42] we propose that functional NNC materials could also be an alternative

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promising catalyst to replace noble metal catalysts in the oxidation of HMF to FDCA.

Herein, we report the first metal-free catalysis of HMF-to-FDCA conversion using ZIF-8-derived NNC nanoparticles as the solid catalysts. Typically, ZIF-8 with controllable particle sizes can be prepared from $Zn(NO_3)_2{\cdot}6H_2O$ and 2-methylimidazole in methanol at room temperature, as modified from a previous report.[36] The prepared ZIF-8 samples were then calcined under N_2 atmosphere at various temperatures from 600 to 900 ^oC to optimize the corresponding specific surface area, pore size, and graphitic carbon framework. Residual zinc was later removed with concentrated HCl prior to an activation process at 150 ^oC in vacuum for 5 hrs. The final samples were named NNC-X, where X represents the carbonization temperatures of 600, 700, 800, and 900 ^oC.

The crystalline structure and surface morphology of the synthesized parent ZIF-8 and NNC materials were characterized with X-ray diffraction (XRD) and scanning electron microscopy (SEM). The XRD patterns of the synthesized parent ZIF-8 and its corresponding NNC nanoparticles were in good agreement with those that were previously reported in literatures (**Fig. S1-S2†**).[36] A broad peak at approximately 2θ = 24^o was observed and its intensity increased with increasing calcination temperature, which indicates the content of graphene in the NNC samples.[43] Additionally, we did not observe any diffraction peaks for Zn or ZnO due to minimal amounts of residual Zn and ZnO in the samples. Another evidence is provided by inductively coupled plasma mass spectrometry (ICP-MS) where the amount of Zn species was measured to be only around 3-8wt% (**Table S1†**). The nanoparticular morphology of the prepared ZIF-8 samples (with an average diameter of around 150-200 nm) is well remained even after calcination at different temperatures and washing with HCl acid, as shown in SEM images (**Fig. S3-S4†**).

The amounts of nitrogen and the porous properties of the synthesized NNC materials were determined by elemental analysis (EA) and nitrogen adsorption/desorption isotherms, respectively. Because our catalysts do not contain any metal, these two properties are important to the catalytic activity of the samples. As shown in **Table S1†**, the nitrogen content decreased from 29.4, 27.7, 21.6 to 15.3wt% as the calcination temperature increased from 600, 700, 800 to 900 ^oC, respectively. This phenomenon can be explained by the collapse of the nitrogen-containing 2-imidazole groups, thus resulting in the reduction of nitrogen content.[44] This phenomenon is also suggested to be the reason for the increased specific surface area of the NNC samples upon carbonization (from 600 to 800 m^2g^{-1} as the calcination temperature increased from 600 to 900 ^oC, respectively) with the same mircopor size of 0.6 nm (**Table. S2 and Fig S5-S8†**).

The bonding configurations of N atom in the NNC samples were examined with X-ray photoelectron microscopy (XPS). As shown in **Fig. S9** and **Table S1†**, the N-1s peak can be deconvoluted to three peaks representing pyridinic-N (N-6) at 398.4±0.2 eV, pyrrolic-N (N-5) at 399.8±0.2 eV, and quaternary-N (N-Q) at 401.1 ±0.2 eV.[45] The pyridinic-N and pyrrolic-N parts (i.e. N-5 + N-6) were predominant (i.e. around 70-95%) in these NNC samples, which has been regarded as electrochemically active sites for various electrochemical applications, such as supercapacitors and Li ion batteries.[35, 36] However, we found that the amount of quaternary-N increased from 4% to 25% as calcination temperature increased from 600 to 900 ^oC, respectively. Quaternary-N (N-Q) specie is regarded as graphitic N, and can serve as a catalytic active site for aerobic oxidation owing to enhanced reaction rate. The detailed mechanism has been explained previously, where sp^2 N-O_2 adducts or oxygen radicals are formed by the adsorption of molecular oxygen on graphitic N or on atomic nitrogen and adjacent carbon, thus becoming active sites for oxidation.[27, 39]

Conversion of HMF to FDCA was conducted using various catalysts. As shown in **Fig. 1a** and **Table S3**, no FDCA production was observed when $Zn(NO_3)_2{\cdot}6H_2O$, 2-methylimidazole, activated carbon (AC), graphite, ZnO, and ZIF-8 were used as catalysts, despite the 100% conversion of HMF to other products. We suggest that this was owing to degradation of HMF in the basic environment of our reaction system. In contrast, FDCA was produced in the presence of the NNC catalysts. For instance, FDCA yields of 11%, 35%, 72%, and 80% were observed with NNC-600, NNC-700, NNC-800, and NNC-900 as the catalyst, respectively. Surprisingly, FDCA yield increased significantly corresponding to the increase of calcination temperature on NNC samples. Because it has been known that the graphitic nitrogen plays a main role for aerobic oxidation,[27] here we analyzed the configuration of nitrogen species in the NNC-X samples.

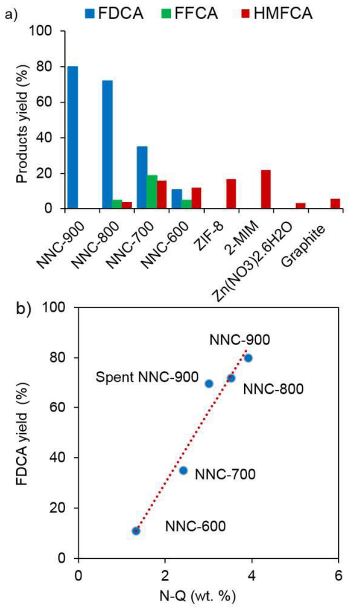

Figure 1: a) The effect of different catalysts on the HMF-to-FDCA conversion. The conversions were 100% for all cases after reaction for 48 hr. b) The relationship between the yield of FDCA and the N-Q atomic percentage on the NNC-X samples. Reaction condition: 0.63 mmol HMF in 10 mL water, 0.2 g catalysts, the molar ratio HMF/K_2CO_3 of 1:3, 100 mL·min^{-1} oxygen at ambient pressure, T=80 ^oC, time =48 hr.

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The FDCA yields versus three types of N-1s were plotted in **Fig. 1b and Fig. S10†**. It is recognized that the FDCA yield is proportional to the amount of the graphitic nitrogen (N-Q) in NNC carbon framework, but there are no connections between N-6 or N-5 and FDCA production. Consequently, we can conclude that graphitic nitrogen is the active site for catalyzing the HMF-to-FDCA conversion. Similar to various aerobic oxidation where N-doped carbons were used as the catalyst,[27, 39] we suppose that the mechanism of the aerobic oxidation is also through a radical process, where the N-Q sites can help generate oxygen radicals for oxidation. To further prove this hypothesis, we added 1,4-benzoquinone as a radical scavenger in the reaction system and found that the production of FDCA was greatly inhibited with the presence of such an anti-radical agent in the system **(Fig. S11†)**.

The reaction route of HMF-to-FDCA conversion using our metal-free catalysts is studied. In general, as shown in **Fig. 2a**, two favourable pathways for aerobic oxidation of HMF to FDCA with the presence of heterogeneous catalysts have been reported.[23, 46, 47] According to the pathway, FDCA can be obtained from FFCA (5-formyl-2-furancarboxylic acid) generated from HMF with either DFF (2,5-diformylfuran) or HMFCA (5-hydroxymethyl-2-furancarboxylic acid) as the intermediates. Here, the kinetics of the HMF-to-FDCA conversion using the NNC-900 catalyst was studied. As shown in **Fig. 2b** and **Fig. S12†,** it can be seen that when HMF was consumed, some intermediates including HMFCA and FFCA were generated and peaked in amounts after eight hours of reaction. As reaction time was further increased, these intermediates were then consumed to produce FDCA. It is worth noting that no DFF intermediate was observed, which indicate that the reaction pathway in this study would be HMF-to-HMFCA-to-FFCA-to-FDCA, as shown in **Fig. 2a**. We also found that the consumption of HMF was accelerated as the reaction temperature was increased, and a 100% consumption of HMF could be achieved within 4 hours at 80 °C (**Fig. 2b**). This result indicated that the HMF-to-HMFCA conversion is the fast step. In contrast, the consumption of HMFCA and FFCA were relatively slow, resulting in a long reaction time (i.e. 48 hours) for the final production of FDCA. These results were in good agreement with a previous report that proposed the FFCA-to-FDCA conversion as the rate-limiting step of HMF-to-FDCA conversion.[48] A higher reaction temperature over 80 °C was not preferred due to a unstable structure of HMF at such a high temperature and basic environment.[48]

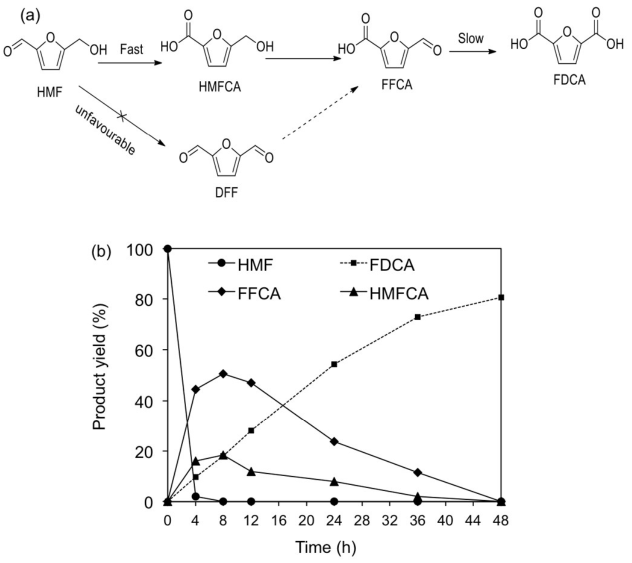

Figure 2: a) possible pathway of converting HMF to FDCA. Reaction condition: 0.63 mmol HMF in 10 mL water, 0.2 g NNC-900 catalyst, the molar ratio HMF/K_2CO_3 of 1:3, 100 mL·min^{-1} oxygen at ambient pressure, reaction temperature of 80 °C; b) The kinetic reaction study.

One of the most important advantages of using heterogeneous catalysts is its recoverability and reusability. The recycle test of the NNC-900 catalyst was thus studied. The aerobic HMF-to-FDCA oxidation was carried at 80 °C using 0.2 g NNC-900 catalyst, in the molar ratio K_2CO_3/HMF of 1:3 (i.e. 0.63 mmol HMF). A flow rate of 100 mL·min^{-1} oxygen gas was purged into the mixture reaction with stir for 48 hr. After each run, the catalyst was separated from the reaction mixture by centrifugation, and washed several times with deionized water and methanol. The sample was then dried under vacuum at 150 °C for 5 h prior to subsequent experiments. As shown in **Fig. 3**, it is found that the catalytic activity of NNC-900 could be reused several times without signifcantly losing activity (i.e. the yield of FDCA was 80% initially and 70% after the fourth run). The spent NNC-900 catalyst was characterized by XRD and XPS, and we found that there was no difference between the crystalline structures but with slightly decreased N-Q amounts (**Fig. S13 and S14† and Table S1†**). It is strongly confirmed that the N-Q configuration of NNC catalysts played an important role for aerobic oxidation of HMF to FDCA. Although nitrogen-doped carbon has been used in HMF oxidation,[27] we conclude that the NNC samples prepared by our method would exhibit higher amounts of the N-Q structure in the carbon framework, resulting in enhanced catalytic performance. The FDCA yield obtained here using a metal-free catalyst is compatible with those previous results using metal-containing catalysts (**Table S4**). Additionally, the effect of the base used in this study (K_2CO_3) on the product distribution and yields was studied. As shown in the **Fig. S15**, the total yields of all products increased when the amount of K_2CO_3 was decreased. Additionally, the highest yield of FDCA was not resulted from the highest amount of K_2CO_3 (i.e. HMF/K_2CO_3 = 1/1) but an optimal ratio of HMF/K_2CO_3 = 1/3. Thus, we

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conclude that an optimized molar ratio of HMF/base is necessary and important.

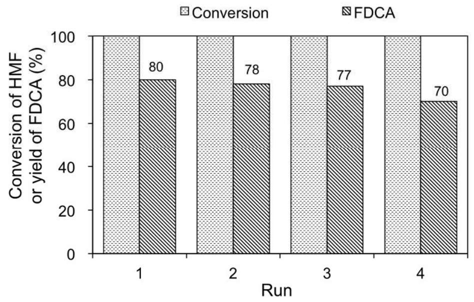

Figure 3: Recycle test of the NNC-900 catalyst. Reaction conditions: 0.63 mmol HMF in 10 mL water, 0.2 g NNC-900 catalyst, the molar ratio HMF/K_2CO_3 of 1:3, 100 mL·min^{-1} oxygen at ambient pressure; reaction temperature and reaction time are 80 °C and 48 hr, respectively.

We have synthesized a new metal-free and nitrogen-containing nanoporous carbon with controllable amounts of nitrogen configuration through a simple pyrolysis of ZIF-8 nanoparticles. Unlike conventional nitrogen-doped carbon materials with low nitrogen-loading density, the ZIF-8 derived nanoporous nitrogen-doped carbon (NNC) enhances the loading amount of nitrogen, especially for the formation of graphitic nitrogen (N-Q) after calcination at 900 °C. The increased N-Q structure in the NNC-900 catalyst was found to be useful for catalyzing aerobic oxidation of HMF to FDCA with a maximum yield of 80% at 80 °C and ambient pressure. The pathway of HMF-to-FDCA conversion is also estimated, where HMFCA and FFCA are the main intermediates. The presenting green, economical and effective heterogeneous NNC catalyst would have much potential in not only biomass conversion but also other oxidation-involved organic reactions.

Acknowledgements

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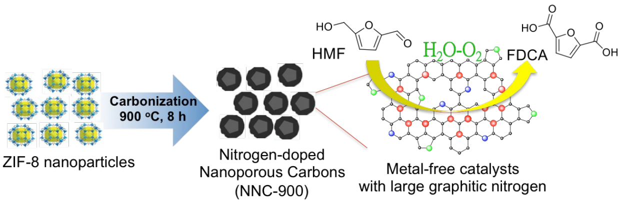
Graphical Abstract

A Metal-Free, High Nitrogen-Doped Nanoporous Graphitic Carbon Catalyst for an Effective Aerobic HMF-to-FDCA Conversion



Chi Van Nguyen, Yu-Te Liao, Ting-Cih Kang, Jeffrey E. Chen, Takuya Yoshikawa, Yuta Nakasaka, Takao Masuda, and Kevin C.-W. Wu*

Nanoporous carbon nanoparticles with high graphitic nitrogen amounts were synthesized and used as a metal-free catalyst for effective HMF-to-FDCA conversion.