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Active Edge Site-Rich Carbon Nanocatalysts with Enhanced Electron Transfer for Efficient Electrochemical Hydrogen Peroxide Production

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Abstract: Electrochemical hydrogen peroxide production enables clean and on-site production of H₂O₂, for which highly active and selective electrocatalysts are of crucial importance. Here, we report a highly efficient, metal-free carbon nanocatalyst that possesses abundant active, oxygenated graphitic edge sites. The edge site-rich nanocarbon catalyst exhibits ~22 times higher activity for H2O2 production than a basal plane-rich carbon nanotube with a H_2O_2 selectivity over 90%. The oxidative treatment further promotes the H_2O_2 generation activity to reach close to the thermodynamic limit. The optimized nanocarbon catalyst shows a very high H_2O_2 production activity, surpassing previously reported catalysts in alkaline media. Moreover, it can stably produce H_2O_2 for 16 hours with Faradaic efficiency reaching 99% and accumulated H₂O₂ concentration of 24 ± 2 mM. Importantly, we find that the heterogeneous electron transfer kinetics of the carbon-based catalyst is closely related to the electrocatalytic activity, suggesting that first outer-sphere electron transfer to O2 is an important step governing the H_2O_2 production rate.

Hydrogen peroxide (H₂O₂) is a green oxidant and one of the most important chemicals in a high industrial demand.^[1] The current production of H₂O₂ depends predominantly on the anthraquinone process.^[2] However, this process involves multistep reactions and separations, which require enormous energy and produce organic wastes. In addition, the use of precious metal catalysts and high-pressure H₂ in the hydrogenation step further increase the production cost.^[2] These drawbacks have triggered the investigation of alternative H₂O₂ production technologies. Electrochemical H₂O₂ generation via the oxygen reduction reaction (ORR) is an appealing method that allows low-cost, green, continuous, and on-site production of H2O2.[3] However, H₂O₂ production from the ORR competes with the complete reduction of O₂ to H₂O via a four-electron (4e⁻) transfer, and thus, requires efficient electrocatalysts that can selectively promote the two-electron ORR.^[4]

Precious metal-based catalysts comprising engineered active sites, such as bimetallic Pt-Hg and Pd-Hg nanoparticles

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and atomically dispersed Pt, have demonstrated excellent catalytic activity and selectivity for H2O2 production.^[5] As lowcost alternative electrocatalysts, metal-free carbon materials have been recently received increasing attention, particularly for H₂O₂ production in alkaline media, where the reaction bath can be directly utilized in some applications.^[6] As pristine carbons are inactive as electrocatalysts, it is important to activate the inert carbon surfaces by creating defective carbon sites and doping heteroatoms. Graphitic carbon edges have been demonstrated to exhibit much superior (electro)catalytic properties than those of basal carbons owing to the modified local electronic structure.^[7] In addition to the enhanced intrinsic reactivity, the edge sites are subject to a facile chemical modification owing to their more reactive nature than basal carbons, enabling a rational design of doped carbons. However, typical graphitic carbons, such as graphene and carbon nanotube (CNT), have a large proportion of basal carbon planes. Moreover, a top-down fabrication of edge-rich carbons from these carbons can cause structural damages, leading to deterioration of the materials properties.

In this work, we demonstrated that a graphitic carbon nanocatalyst with exposed abundant edge sites could catalyze H_2O_2 production with a ~22 times higher mass activity than that of a basal plane-rich CNT. Additional oxidative treatments of the graphitic carbon nanocatalyst could preferentially generate active oxygenated species at the carbon edge, which further promoted the catalytic activity for H2O2 production reaching close to the thermodynamic limit. It was revealed that the activity improvement through the edge-rich design and oxidative promotion could be correlated to the heterogeneous electron transfer (ET) rate, thereby suggesting that the first outer-sphere ET to O₂ was a critical step determining the H₂O₂ production rate. The optimized nanocarbon catalyst stably produced $H_2O_2\ \text{for}\ 16$ h without activity loss. In addition, a significant H₂O₂ accumulation of 24 ± 2 mM was achieved, demonstrating its tremendous potential for direct utilization in areas such as bleaching and acidic waste treatments.

Catalyst preparation consisted of the infiltration of an aromatic carbon precursor into a mesoporous silica with a hexagonal mesostructure, graphitization, and removal of the silica template. The mesopores in the silica template acted as nanoreactors within which confined graphitization generated nano-sized graphite, whereas its silica frameworks transformed into mesopores after the removal of the template. The resulting graphitic ordered mesoporous carbon (GOMC) showed a CMK-3-like mesostructure with graphitic frameworks.^[8] The GOMC retained the parental hexagonal periodic mesostructure of the silica template as revealed by the small-angle X-ray diffraction (XRD) patterns (Figure S1). The transmission electron microscopy (TEM) image of the GOMC shows a hexagonal array of carbon nanorods with a diameter of ~9 nm (Figure 1a).

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Figure 1. a) Side-view TEM image of the GOMC (inset: top-view TEM image, scale bar = 25 nm). High-resolution TEM image of b) the GOMC and c) CNT with schematic models for each carbon. d) XRD patterns e) Raman spectra, and f) C 1s XPS spectra of the GOMC and CNT.

The high-resolution TEM image exhibits a vertical stacking of slightly wrinkled graphitic nanosheets with nanopores present between the graphitic rods (Figure 1b). This characteristic nanostructure of the GOMC enables a high degree of utilization of the graphitic edges in the carbon nanorods. For comparison, we prepare an acid-leached CNT mainly comprising of graphitic basal planes (Figure 1c). We note that acid-leaching is an important step for removing the iron impurities from the original CNT, which can otherwise catalyze the Fenton reaction causing underestimation of the H_2O_2 production capability of the CNT.[6d,9] The XRD pattern of the GOMC exhibits a broader and negatively shifted peak for the (002) plane of graphite than that of the CNT, indicating a smaller grain size and larger interplanar distance in the GOMC (Figure 1d). The defective nature of GOMC was verified by Raman spectroscopy. The Raman spectrum of the GOMC shows a broad D band peak, indicative of the presence of a large portion of defective carbons, along with a sharp G band (Figure 1e).^[10] In contrast, the CNT exhibits sharp Raman peaks for both the D and G bands. Carbon 1s Xray photoelectron spectroscopy (XPS) further suggests that the GOMC has more defective carbon sites than those in the CNT, evidenced by the wider peak for sp² C (Figure 1f).^[6e] The higher XPS signals for the oxygenated carbon species in the GOMC are presumably due to O-doping, which may originate from the silica template (Figure S2). The oxygen content of the catalysts determined by combustion analysis were 2.4 wt% for the GOMC and 1.0 wt% for the CNT.

The electrocatalytic performances of the GOMC and CNT for H₂O₂ production via ORR were examined in 0.1 M KOH using the rotating ring disk electrode (RRDE) technique. The polarization curves of the catalysts presented in Figure 2a reveal that the GOMC exhibits a much higher ORR activity than that of the CNT, indicated by a positive shift of the onset potential by 65 mV in the GOMC (0.78 V vs. reversible hydrogen electrode; RHE). The kinetic current of the GOMC for H_2O_2 production at 0.75 V is 22 times higher than that of the CNT (Figure 2b). In addition, the GOMC and CNT have similar Brunauer-Emmett-Teller (BET) surface areas of 279 and 187 m² g⁻¹, respectively (Figure S3 and Table S1). The most dramatic structural difference between the GOMC and CNT is the density of edge sites. Therefore, the graphitic carbon edges in the GOMC seem to be the major active sites for the electrochemical H_2O_2 generation. Furthermore, the GOMC exhibits a high selectivity for H_2O_2 of >90% over the entire potential range investigated, whereas the CNT is less selective than the GOMC, particularly close to the onset potential (Figure 2c). The Koutecky-Levich analyses also indicate a high two-electron (H₂O₂) selectivity of the GOMC (Figure S4).

We next explored the possibility of further promoting H₂O₂ production via doping of the oxygenated species in the highperforming GOMC. For this purpose, the GOMC was acidtreated in nitric acid, and the oxidation degree was controlled by adjusting the treatment time from 1 hour to 8 hours.

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Figure 2. a) Polarization curves of the GOMC and CNT for the ORR (solid lines) and H_2O_2 production (dashed lines) measured in O_2 -saturated 0.1 M KOH at an electrode rotation speed of 1600 rpm and b) corresponding Tafel plots for the H_2O_2 production. The peroxide current was calculated by the correction of the ring current with the collection efficiency of the RRDE. c) H_2O_2 selectivity determined using the RRDE method.

The resulting catalysts were denoted as o-GOMC-X (X: oxidation time). The oxygen content was increased from 2.4 to 10.1-16.2 wt% after oxidation for 1-8 hours (Figure S5). The edge-rich structure of the GOMC could facilitate the oxygen incorporation into carbon, leading to O content increase by 7.7 wt% after just 1 h of oxidative treatment. We noted that despite

the generation of abundant O species after the acid-treatment, the periodic nanoporous structure and graphitic nature of the GOMC were retained. This is indicated by the similar XRD patterns and N₂ physisorption isotherms of the GOMC and o-GOMCs (Figures S3, S6, and Table S1). Incorporation of O atoms into the carbon is verified via XPS analyses, wherein the intensities of C-O, C=O, and O-C=O increase on increasing the treatment duration (Figure S7). It is noteworthy that the C 1s XPS intensity for the $\pi\text{--}\pi^{\star}$ shake-up (Figure S7a) and Raman spectra (Figure S8) of the o-GOMCs are preserved even after the acid treatments. The Raman and XPS results indicate that O atoms are mainly incorporated at the carbon edge sites. We note that the O dopants at the carbon edge were suggested as active sites for the two-electron ORR.[6d,6e] Therefore, this unique structural feature of o-GOMCs is expected to be favorable for electrochemical H₂O₂ production.

The ORR activities and H₂O₂ selectivities of the o-GOMCs are presented in Figure 3. The oxidative modification of the GOMC can further improve the ORR activity, as indicated by the positively shifted polarization curve of o-GOMC-1 compared with GOMC (Figure 3a). It should be noted that the onset potential of 0.81 V (vs. RHE) is almost close to the thermodynamic limit of the two-electron ORR.^[6e] Therefore, the o-GOMC-1 catalyst with abundant O-containing carbon edges is highly active for electrochemical H₂O₂ production. However, as the oxidation time is increased, the ORR activity deceases (Figure 3a). This indicates that the ORR activity of the GOMC follows a volcanolike trend with the O-doping level. In contrast, the acid-treatment does not affect the reaction mechanism, as revealed by the similar H₂O₂ selectivities and Tafel slopes (Figure 3b and Figures S9, S10). Therefore, O-doping at the carbon edge boosts the reaction rates while maintaining a high selectivity of over 90%.

The long-term stability of o-GOMC-1 was tested via chronoamperometry (average of three repetitions) at 3 mA for 16 h using an H-type electrochemical cell. After a prolonged operation, ~96% of the initial current is retained, demonstrating its excellent stability (Figure 3c). To determine the Faradaic efficiency for H₂O₂ production, the amount of H₂O₂ in the tested electrolyte was analyzed using iodometric titration.^[6e] The quantification result indicates that the H₂O₂ Faradaic efficiency of o-GOMC-1 is 99%, which corresponds to H₂O₂ accumulation of 24 ± 2 mM. This concentration is sufficiently high for direct usage in some applications such as bleaching.^[2,4b]

It is also worthwhile to note that the o-GOMC-1 catalyst did not deteriorate in the presence of the oxidizing chemical (i.e., H_2O_2) and did not further catalyze the reduction or decomposition of H_2O_2 during the reaction. This can be ascribed to the robust graphitic carbon framework, which is, however, active owing to the presence of oxygenated edges. These excellent electrochemical performances of o-GOMC-1 surpass those of most the other reported carbon nanocatalysts in alkaline media (Figure S11 and Table S2).^[6]

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Figure 3. a) Polarization curves of the GOMC and o-GOMCs for the ORR (solid lines) and H_2O_2 production (dashed lines) measured in O_2 -saturated 0.1 M KOH at an electrode rotation speed of 1600 rpm and b) corresponding H_2O_2 selectivity determined using the RRDE method. The peroxide current was calculated from the correction of the ring current with the collection efficiency of the RRDE. c) Chronoamperometic response of o-GOMC-1 in O_2 -saturated 0.1 M KOH in an H-type electrochemical cell.

The enhanced ORR activity of doped carbon materials to H₂O₂ production has been predominantly substantiated by computational methods that provide free energies of the reaction intermediates, which are, however, hardly determined empirically.^[6d,6g] We here provide the experimental evidence elucidating the volcano-like ORR activity trends of the nanocarbon catalysts. Tafel slopes ranging from 48 to 64 mV dec⁻¹ are obtained for the GOMC, o-GOMCs, and CNT, where the Tafel slope close to 59 mV dec⁻¹ suggests that the ORR rate is limited by the first ET to the O_2 molecule under the Temkin adsorption conditions (Figure S9).[11] This kinetic insight gives rise to an assumption that the ET ability of the carbon is a prime determinant of the ORR activity. To evaluate the ET rate of the carbons, an outer-sphere redox system of [Fe(CN)6]3-/4- was exploited. Cyclic voltammograms, independently measured at least five times, show redox peaks, and the interval between the peaks determines the observed heterogeneous electron rate constant (k_{obs}^{0}) obtained from the Nicholson equation (see the Supporting Information for details, Figure S12 and Table S3).^[12] The k_{obs}^0 values and ORR mass activity (at 0.75 V vs. RHE) show a similar trend in which the maximum appears at approximately between 5-8 wt% of the O-doping level (Figure 4a). The k_{obs}^0 values plotted versus the mass activities clearly display a linear relationship (Figure 4b). The similar trend and correlation are also clearly seen in the specific activity-based comparison (Figure S13). This correlation reveals that the ET capability governs the ORR activity of the doped carbon materials and further supports that the rate-determining step is the first outer-sphere ET to O₂. The ability of the catalyst for the ET was further confirmed by electrochemical impedance spectroscopy (EIS), which provides the charge transfer resistance (R_{ct}). The Nyquist plots of the EIS spectra at 0.75 V (vs. RHE) show smaller semicircles for o-GOMC-1 and o-GOMC-3 than that for GOMC (Figure 4c). Smaller semicircles indicate a lower R_{ct} , and thus, a more facile ET in o-GOMC-1 and o-GOMC-3 than that in the GOMC. In contrast, o-GOMC-8 and the CNT exhibit higher R_{ct} than that for the GOMC. The EIS analysis results are highly consistent with the k_{obs}^0 and ORR activity trends (Figure S14 and Table S4). The reciprocal of R_{ct} , related to the charge transfer rate, also matches well with the k_{obs}^0 and ORR activity (inset of Figure 4c).

In conclusion, we demonstrated highly selective and active electrochemical H_2O_2 production via the ORR using carbon nanomaterials that are rich in oxygenated carbon edges. The experimental evidence suggested that the faster heterogeneous outer-sphere ET was responsible for the higher ORR activity of the oxidized and edge-rich carbon nanocatalysts. This kinetic insight into the ET, which has thus far been largely neglected, was revealed to play a crucial role, providing an important guideline for the design of carbon-based electrocatalysts. Furthermore, the large H_2O_2 accumulation by the GOMC and its excellent stability during the prolonged operation shed light on the feasibility of our carbon nanocatalysts in the electrochemical H_2O_2 production technology.

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Figure 4. a) Trends of the mass activity at 0.75 V (vs. RHE) (bottom) and heterogeneous ET rate constant (top) with the change in the O content. b) Relationship between the mass activity at 0.75 V (vs. RHE) and heterogeneous ET rate constant. c) Nyquist plots of the GOMC, o-GOMCs, and CNT measured at 0.75 V (vs. RHE) with an electrode rotation of 1600 rpm in O_2 -saturated 0.1 M KOH (inset: trends of the reciprocal of the charge transfer resistance value with the O content).

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Keywords: hydrogen peroxide • carbon nanomaterial • electrocatalyst • oxygen reduction • electron transfer

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