Electrospun Cobalt-ZIF Micro-Fibers for Efficient Water Oxidation at Unique pH Conditions

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This file contains 25 pages where the information on the reagents and instruments used, schematic representations, XRD, SEM, XPS and electrochemical studies are given respectively.
Reagents and Instruments

Cobalt chloride hexahydrate (CoCl$_2$. 6H$_2$O) and polyacrylonitrile (PAN) were procured from sigma-aldrich. 1-methylbenzimidazole and dimethylformamide (DMF) were purchased from Alfa-aesar. For electrospinning, ESPIN from PECO was used. The electrochemical analyzer AUT-302N. FRA32M was used for all electrochemical characterizations. Hg/HgO reference electrode, Hg/HgSO$_4$ reference electrode and Pt counter electrode were purchased from CH instruments and CC working electrode purchased from Alfa-Aesar. DI water is used during the course of synthetic process. The Co-ZIF microfibers prepared at different conditions were characterized with the FE-SEM instrument (SUPRA 55VP Carl Zeiss) with a separate EDS detector connected to that instrument. Color mapping and Energy Dispersive X-ray Spectroscopy (EDS) analysis were done with the aid of FESEM instrument. The XRD analysis was done with a scanning rate of 5° min$^{-1}$ in the 2$\theta$ range 10-90° using a Bruker X-ray powder diffractometer (XRD) with Cu K$_\alpha$ radiation ($\lambda = 0.154$ nm). X-ray photoelectron spectroscopic (XPS) analysis was performed using a Theta Probe AR-XPS system (Thermo Fisher Scientific, UK). Electrochemical analyzer AUT-302N. FRA32M was used for the entire OER and related studies.
Scheme S1. Synthesis procedure of Co-ZIF and precursor material for Electrospinning.
**Scheme S2**: Electrospinning method utilized for the formation of Co-ZIF microfibers.
Figure S1: a) is the XRD pattern of Co-ZIF powder and b) is the XRD pattern of microfibers of Co-ZIF-RT.
Figure S2: (a-d) are the FESEM images of Co-ZIF-RT at different magnifications that showed the exact fiber formation at room temperature.
Figure S3: (a and b) are the SEM images of Co-ZIF-RT showing fiber morphology and (c) is the corresponding EDS spectrum.

Carbon- 70.22%
Nitrogen- 22.56%
Chlorine- 3.43%
Cobalt- 1.77%
Oxygen- 2.02%
Figure S4: (a, b) are the FESEM images of Co-ZIF-350-N$_2$ (c-f) are corresponding mapping of C, N, Co, O respectively.
Figure S5: (a-c) are the TEM images of Co-ZIF-550-N$_2$ from the low to high magnification and (d) is the corresponding SAED pattern.
Figure S6: (a-d) are the XPS spectrum of corresponding elements of Co 2p, C 1s, N1s and O 1s in Co-ZIF-RT respectively.
Figure S7: (a-d) are the XPS spectrum of corresponding elements of Co 2p, C 1s, N1s and O 1s in Co-ZIF-350-N$_2$ respectively.
Figure S8: Polarization curves acquired in 0.5 M H$_2$SO$_4$ without iR compensation.
Figure S9: Polarization curve acquired at 1 mV/sec for Co-ZIF-550-N$_2$ after the cycling test for 250 cycles at 200 mV/sec.
Figure S10: (a) is the polarization curve acquired at 1 mV/sec for Co-ZIF-550-Air and (b) is the corresponding chronoamperometry study.
Figure S11: (a and b) are the observed CV at different scan rates like 10, 30, 60 and 120 mV /sec and (c) is the corresponding $C_{dl}$ values at acid medium.
Figure S12: a) is the XRD pattern of Co-ZIF-550-N$_2$ observed after OER study in 0.5 M H$_2$SO$_4$. 
Figure S13: Post OER SEM images (a and b) of Co-ZIF-550-N₂ at different places.
**Figure S14:** (a-d) are the post OER XPS spectrum of corresponding elements of Co 2p, C 1s, N1s and O 1s in Co-ZIF-550-N2 in 0.5 M H2SO4 respectively.
**Figure S15:** Polarization curves acquired in 1 M KOH without iR compensation.
Figure S16: Polarization curve acquired at 1 mV/sec for Co-ZIF-350-Air after the cycling test for 500 cycles at 200 mV/sec.
Figure S17: (a) is the polarization curve acquired at 1 mV/sec for Co-ZIF-550-Air and (b) is the corresponding chronoamperometry study.
Figure S18: (a and b) are the observed CV at different scan rates like 10, 30, 60 and 120 mV sec\(^{-1}\) and (c) is the corresponding \(C_{dl}\) values at alkaline medium.
**Figure S19:** a) is the XRD pattern of Co-ZIF-350-Air observed after OER study in 1 M KOH.
**Figure S20**: Post OER SEM images (a and b) of the Co-ZIF-350-Air at different places.
Figure S21: (a-d) are the post OER XPS spectrum of corresponding elements of Co 2p, C 1s, N 1s and O 1s in Co-ZIF-350-Air in 1 M KOH respectively.