Supporting information

Three dimensional Ni_4O_4-cubane metal organic framework as a high-performance urea oxidation electrocatalyst

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Calculation method

Determination of Tafel slope

Tafel slope was calculated by overpotential (η) and current density (j) by using Tafel equation.[1]

$$\eta = b \log j + a$$

TOF

Turnover frequency (TOF) values of different samples were calculated according to the equation:[2]

$$TOF = \frac{j\ X\ A}{6\ X\ F\ X\ n}$$

Where j is the current density at a given overpotential, A is the surface of the electrode, F is the Faraday constant, and n is the number of moles of metal on the electrode.

Mass activity

$$\text{Mass activity} = j/m^{3}$$

Mass activity (A/g) value was calculated from the electrocatalyst loading m (0.32 mg/cm^2) and the measured current density j (10 mA/cm^2 at 1.24 V vs. RHE)

Determination of electrochemical double-layer capacitance (C_{dl})

For electrochemical double-layer capacitance (C_{dl}) determination, the scan rate dependent CV measurements were carried out between 2 mV/s to14 mV/s over a small window where no Faradaic current was observed (1.065 – 1.165 V). The electrochemical double layer capacitance (C_{dl}) was extracted from $i_c = \nu \times C_{dl}$, where ν is the scan rate and i_c is the current density for the specific curve at 1.11 V.[1]

ECSA

The electrochemical active surface area (ECSA) is calculated as[2]

$$\text{ECSA} = C_{dl}/C_s$$

Where C_{dl} is double layer capacitance and C_s is the specific capacitance ($C_s = 60\ \mu F\ cm^{-2}$)

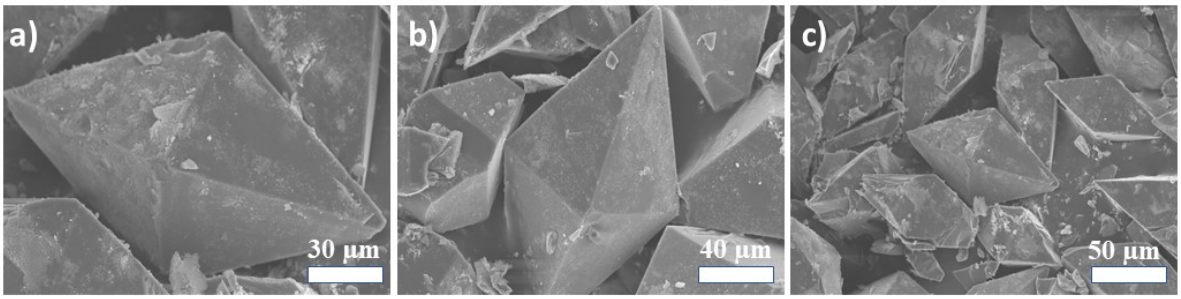

Fig. S1 SEM image of **MOF 1** at different resolutions a) 30 µm b) 40 µm c) 50 µm

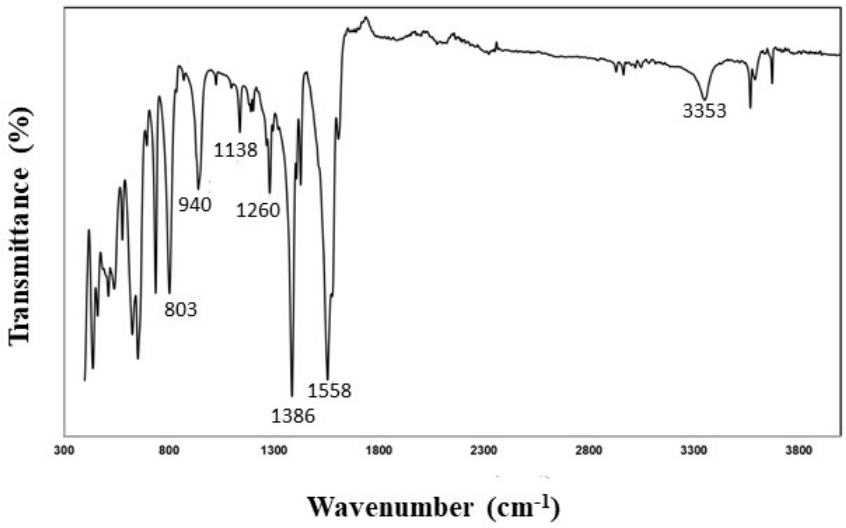

Fig. S2 FT-IR spectrum of **MOF 1**[4]

Thermogravimetric Analysis

Thermogravimetric analyses were performed under nitrogen flux using a Mettler Toledo TGA/SDTA 851 at a heating rate of 10 °C min^{-1} to assess the thermal stability of **MOF 1** (Fig. S2). **MOF 1** is thermally stable up to 380°C. An initial weight loss (ca. 5%) starting at 150 °C is due to loss of one water molecule coordinated to Ni3 (calc. 5.06%) in the framework. At higher temperatures the mass remains constant, followed by sudden decrease in the weight, suggesting the onset of decomposition around 380°C with a total weight loss of 55.3% (calc. 57.98%), resulting in simple oxides (NiO).

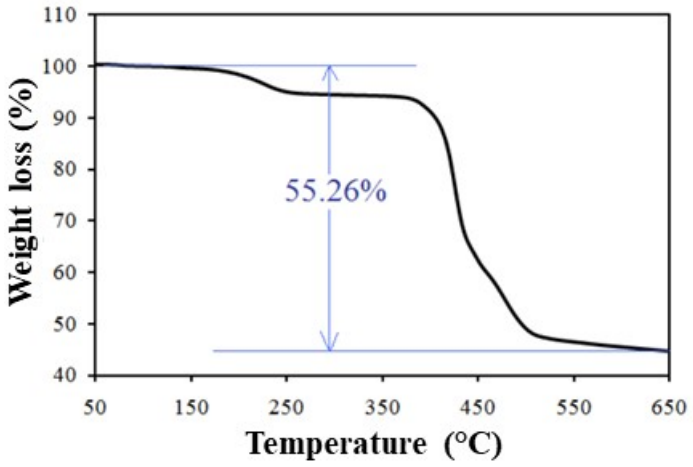

Fig. S3 Thermogravimetric analysis of **MOF 1**[4]

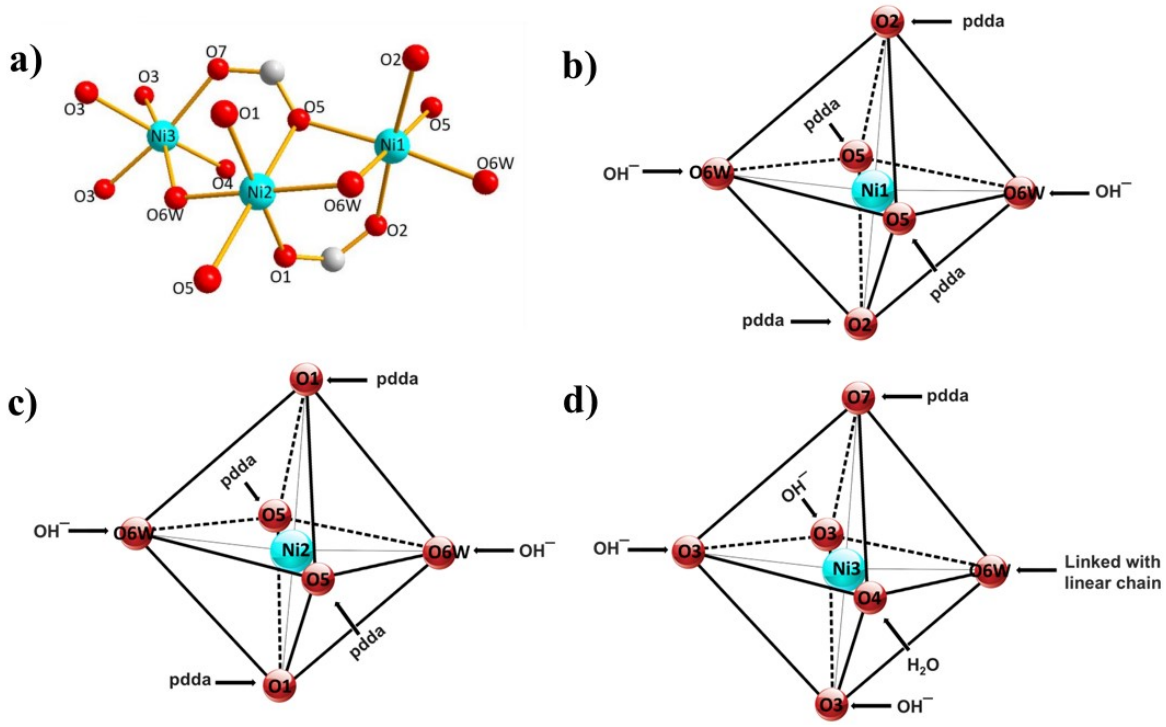

Fig. S4 a) Coordination environment of all three Ni b) Ni1 c) Ni2 d) Ni3

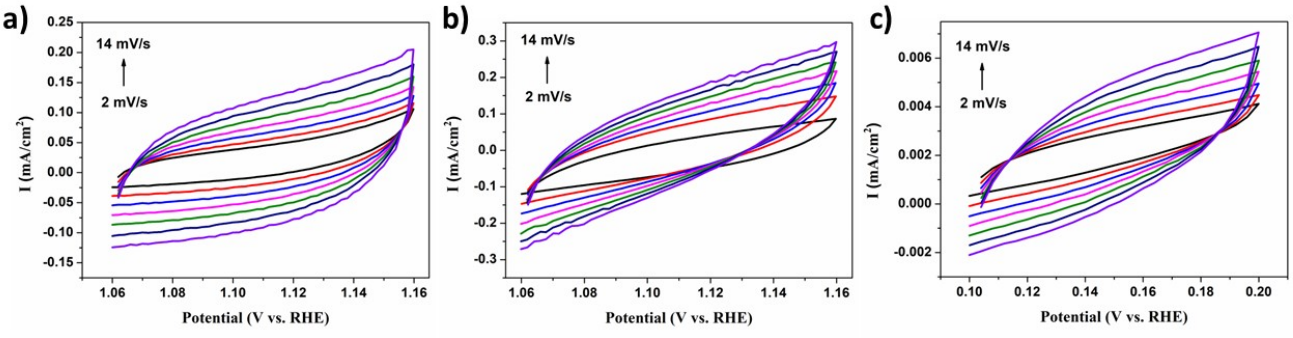

Fig. S5 Cyclic voltammograms in non-faradaic region with incremental 2 mV/s sweep rate a) Pt/C b) $Ni(OH)_2$ c) Bare FTO

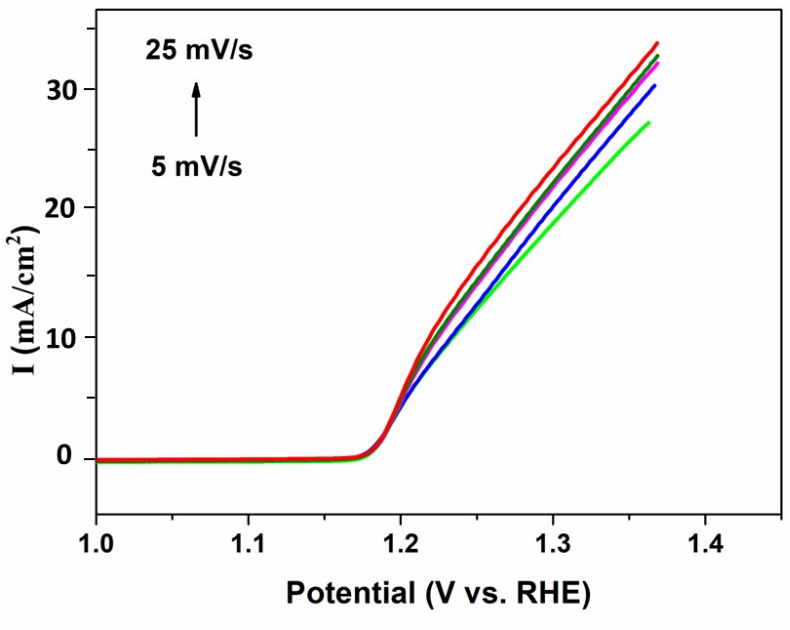

Fig. S6 LSV of **MOF 1** at varied scan rates (5 mV/s to 25 mV/s)

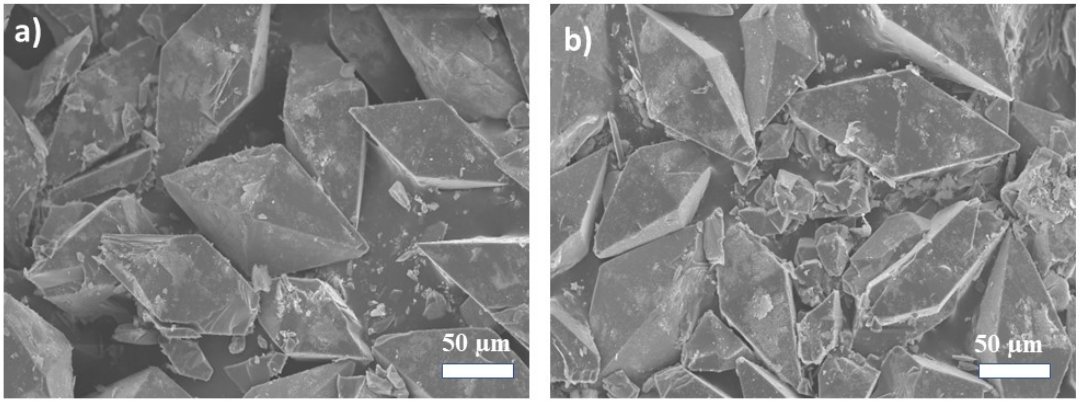

Fig. S7 SEM image of **MOF 1** a) before stability test b) after 17 h continuous test

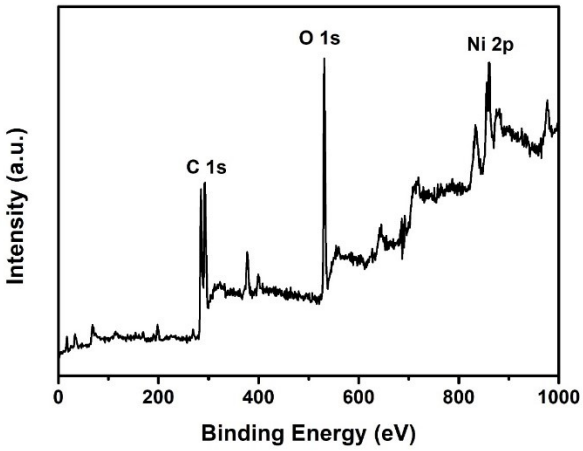

Fig. S8 XPS survey spectrum of **MOF 1**

References

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2. Z. Gao, Y. Wang, L. Xu, Q. Tao, X. Wang, Z. Zhou, Y. Luo, J. Yu and Y. Huang, *Chem. Eng. J.* 2022, **433,** 133515.
3. J. Huang, J. Chen, T. Yao, J. He, S. Jiang, Z. Sun, Q. Liu, W. Cheng, F. Hu and Y. Jiang, *Angew. Chem. Int. Ed.* 2015, **127,** 8846.
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