

Supporting Information

Bifunctional Electrochemical OER and HER Activity of Ta₂O₅ Nanoparticles Over Fe₂O₃ Nanoparticles

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Instrumentation

Powder X-ray diffraction (PXRD) pattern was obtained on a Bruker D8 Advances instrument using Cu-K α ($\lambda = 1.5406 \text{ \AA}$) radiation in the 2θ range from 10° to 80° with an acceleration voltage of 40KV. X-ray photoelectron spectroscopy (XPS) measurements were carried out with a Kratos Axis Ultra system, equipped with a monochromatic AlK α X-ray source. The survey spectra were measured with 80 eV pass energy and 1 eV step size whereas with the high-resolution spectra 20 eV pass energy and 100 mV step size was used. All XPS measurements were performed using the charge neutralizer. FESEM instrument was used to perceive scanning electron microscopy (SEM) images (Carl Zeiss, Voltage = 20 kV). All electrochemical measurements were performed with Metrohm Autolab (M204 multichannel potentiostat galvanostat using Nova 2.1.4 software).

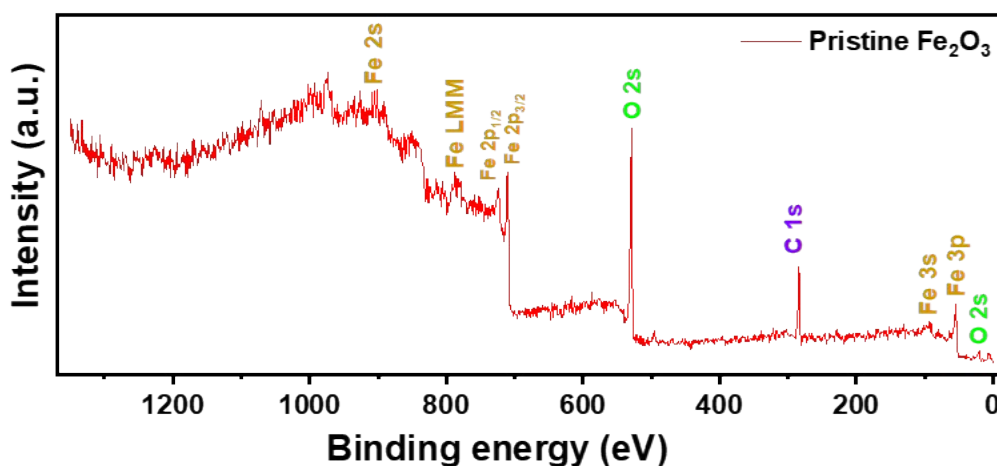


Figure S1. XPS survey spectra of pristine Fe₂O₃

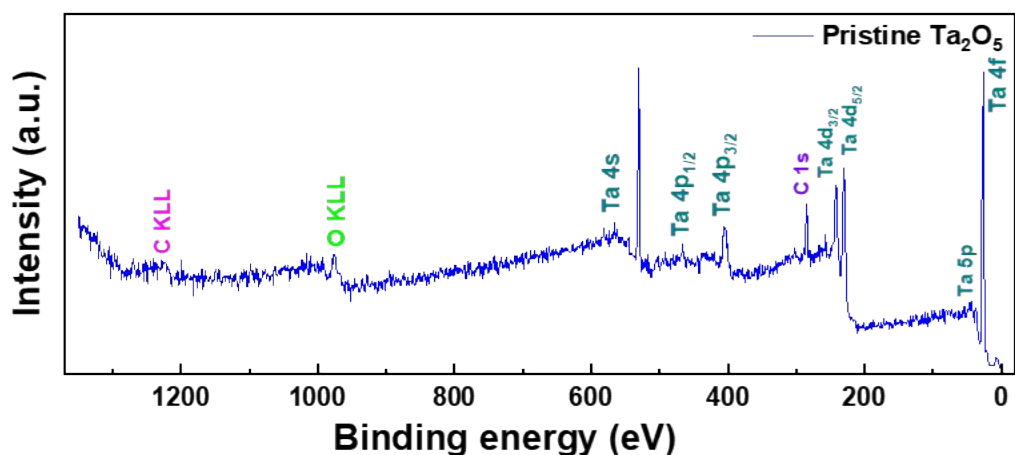


Figure S2. XPS survey spectra of pristine Ta₂O₅

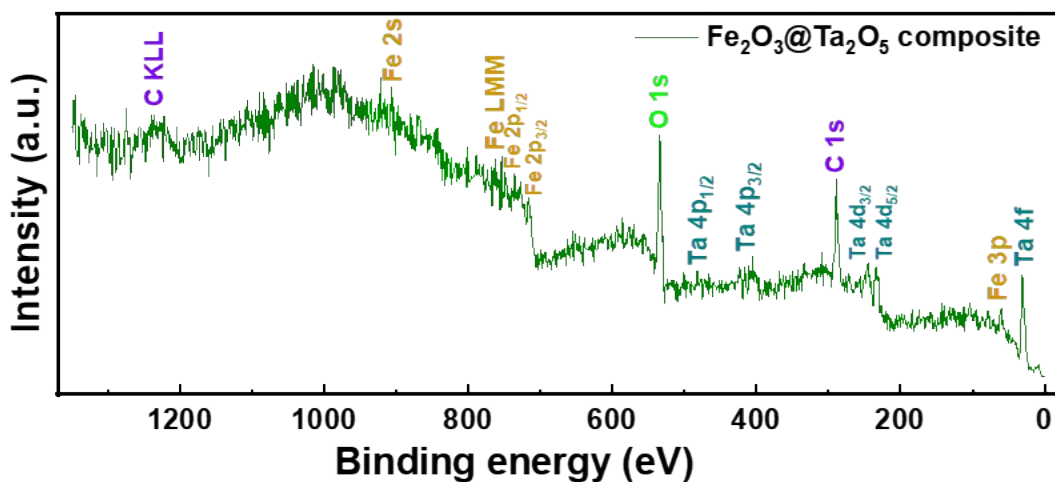


Figure S3. XPS survey spectra of Fe₂O₃/Ta₂O₅ composite.

Preparation of electrodes and detail of electrochemical studies

To prepare the working electrode, 3 mg of catalyst was taken in a 1.5 mL micro centrifuge tube and dissolved with a mixed solution of 280 μ L of double distilled water (18.2 Milli-Q) and 200 μ L of ethanol. Afterwards, the resulting mixture was sonicated for 30 minutes prior to addition of 20 μ L 5% Nafion solution and further sonication was continued using GT-sonic ultrasonicator for another 15 min to obtain a homogeneous ink. The as-prepared 5 μ L catalyst ink was drop-coated

onto the glassy carbon (GC) electrode (having diameter 3 mm) and then it was left for drying in desiccator (under mild vacuum) for overnight. The resulting electrode was used as working electrode (WE) for the study of oxygen evolution reaction (OER) and hydrogen evolution reaction (HER) and the mass loading of catalysts was calculated to be 0.424 mg cm⁻².

All the electrochemical measurements were carried out by using Metrohm Autolab (Multichannel M204) connected to a standard three-electrode electrochemical cell using Nova 2.1.4 software. For the electrochemical characterizations the samples were coated GC electrode as working electrode. Platinum (Pt) electrode and graphite rod were used as a counter electrode for OER and HER studies, respectively. Whereas Ag/AgCl (3M KCl) and Hg/HgO electrodes were taken as reference electrode for HER, and OER studies, respectively. All the experiments related to OER were carried out in 1M KOH (pH 13.8), and HER studies were performed in 0.5 M H₂SO₄ solution prepared using double distilled water. The linear sweep voltammetry (LSV) was carried out with a scan rate of 10 mV s⁻¹ for both OER and HER studies and cyclic voltammetry (CV) experiments were carried in the potential range of 0.2 V to 0.3 V (vs. Hg/HgO) with a scan rate from 20 mV s⁻¹ to 100 mV s⁻¹. The LSV cycle stability (3000 cycle) of the Fe₂O₃/Ta₂O₅ catalyst was studied with a constant scan rate of 50 mV s⁻¹. The electrochemical impedance spectroscopy (EIS) was carried out over the frequency range of 100 kHz to 0.1 Hz. All the potentials were converted to RHE using the following Nernst equation for HER activities.

$$E_{(\text{RHE})} = E_{(\text{Ag/AgCl})} + 0.059\text{pH} + E_{\text{Ref}}$$

All the potentials were converted to RHE using the following Nernst equation for OER activities.¹

$$E_{(\text{RHE})} = E_{(\text{Hg/HgO})} + 0.059\text{pH} + E_{\text{Ref}}$$

Calculation of electrochemical surface area

The electrochemically active surface area (ECSA) is proportional to the electrochemical double layer capacitance (C_{dl}) and the ECSA can be calculated using the following equation.

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

Where C_s is the specific capacitance of flat working electrode and its value is 40 μF cm⁻² ECSA for the flat electrode.^{2,3} The double-layer capacitance (C_{dl}) is calculated from the slope of anodic

current density (J_a), cathodic current density (J_c) vs scan rate plot. The slope is directly proportion to the C_{dl} value.

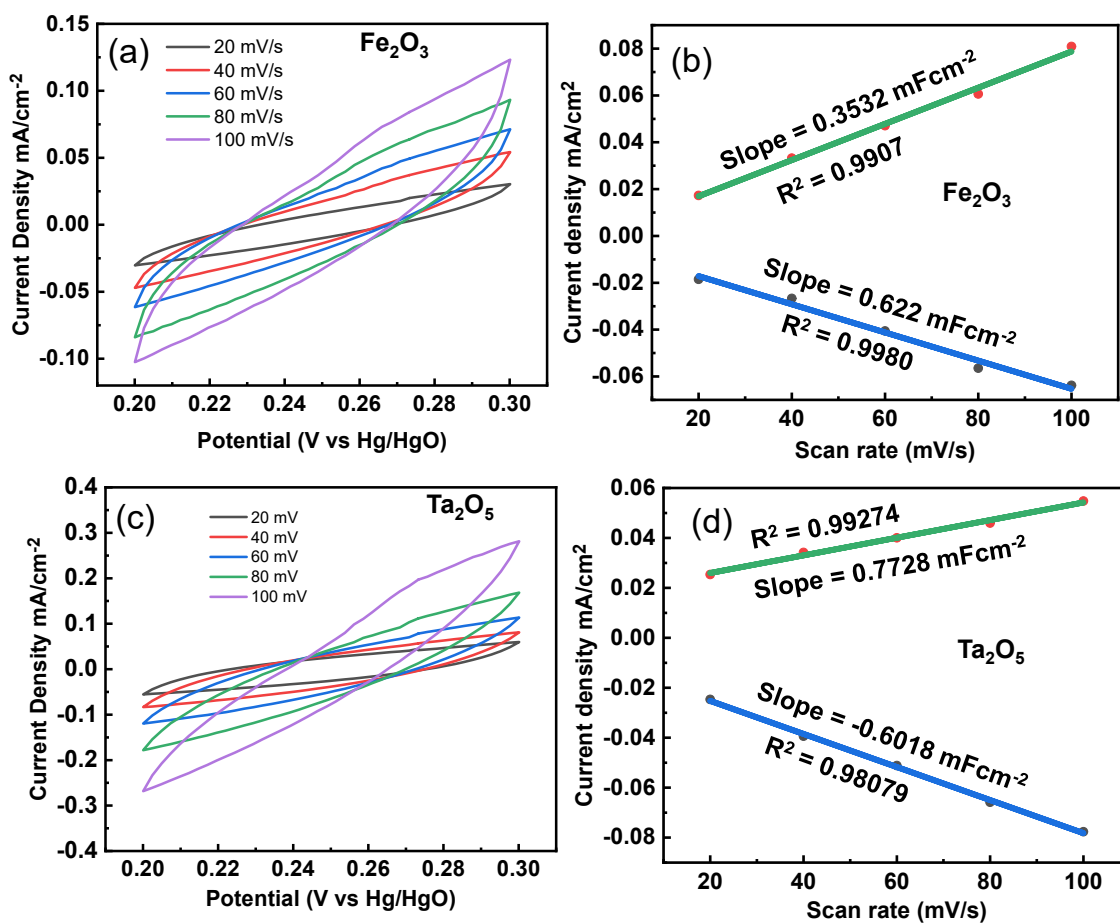


Figure S4. Cyclic voltammetry curves of (a) pristine Fe₂O₃ (c) pristine Ta₂O₅ (b-d) are their corresponding plot of J_a and J_c against scan rate for the determination of double layer capacitance (C_{dl}) of the catalysts, respectively.

Mass activity

Mass activity was calculated using the following equation.

$$\text{Mass activity (A g}^{-1}\text{)} = \frac{j \text{ (A cm}^{-2}\text{)}}{m \text{ (g cm}^{-2}\text{)}}$$

Where, j = current density,

m = mass loading (here $m = 5 \times 10^{-5}$ mg)

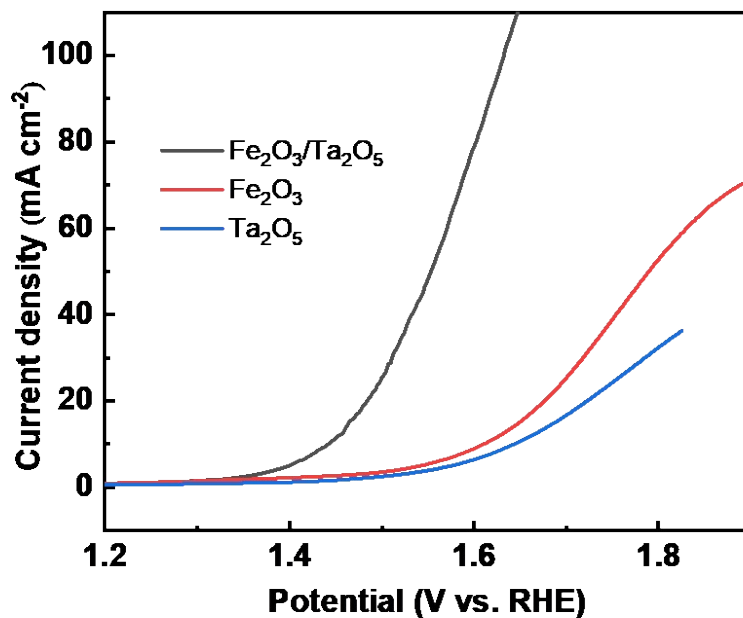


Figure S5. LSV plots representing the mass activity (A/g) of Fe₂O₃, Ta₂O₅ and composite Fe₂O₃/Ta₂O₅ catalysts.

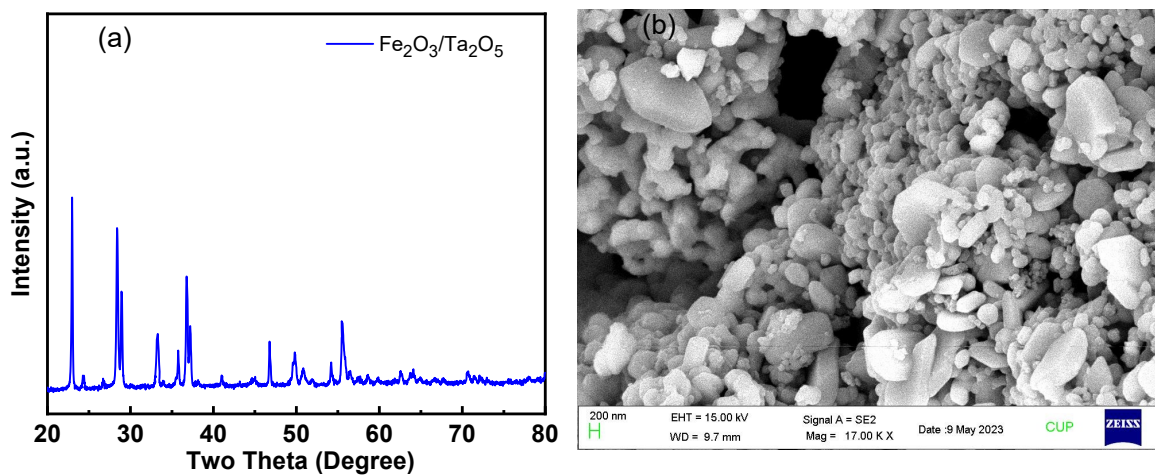


Figure S6. Post catalytic study of composite Fe₂O₃/Ta₂O₅ (a) XRD and (b) FE-SEM.

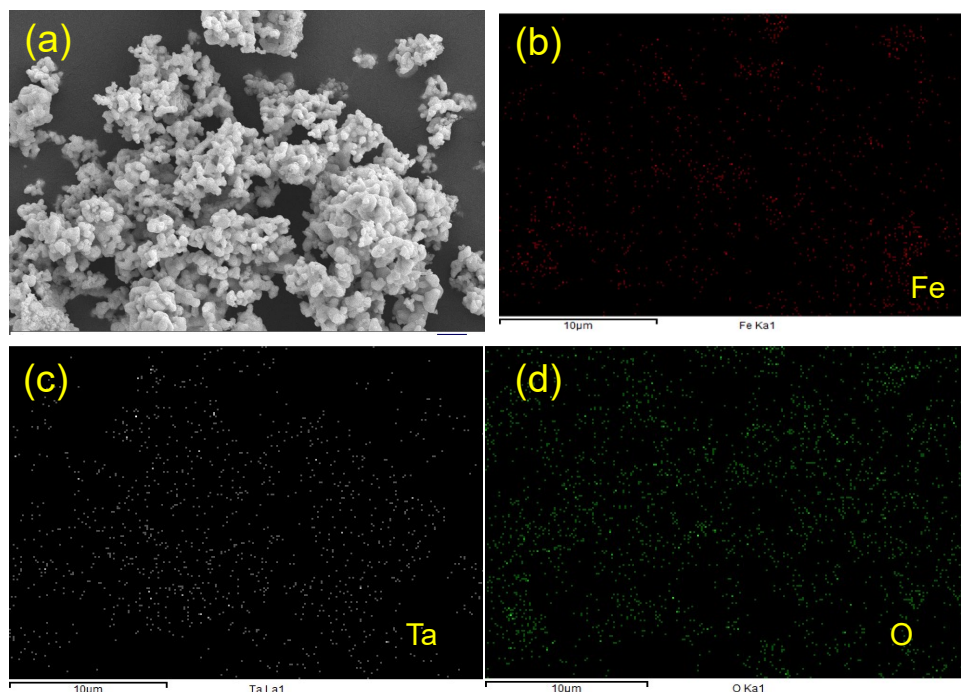


Figure S7. Post catalytic elemental mapping of composite $\text{Fe}_2\text{O}_3/\text{Ta}_2\text{O}_5$.

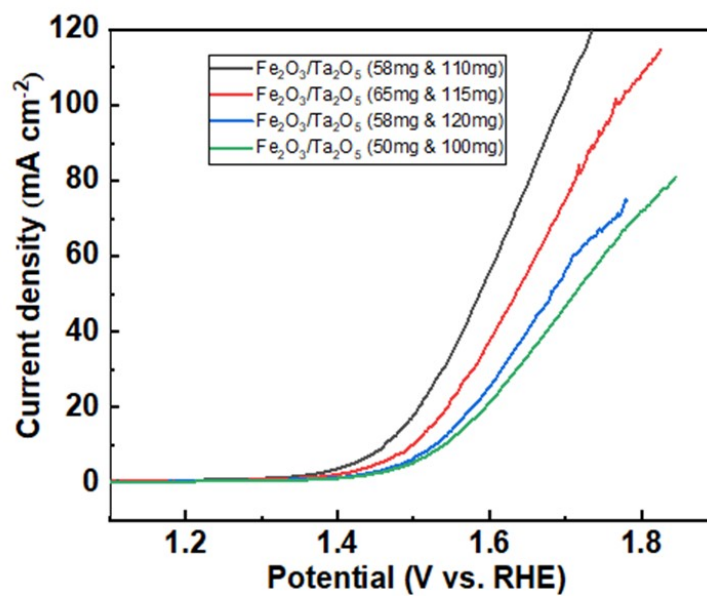


Figure S8. LSV plots representing the activity of different composition (by weight).

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