## **Supporting Information for**

## Bismuth-Rich Bismuth Oxyiodide Microspheres with Abundant

## Oxygen Vacancies as an Efficient Photocatalyst for Nitrogen Fixation

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## **Supplementary figures**





The BiOI X-ray diffraction (XRD) patterns are shown in Fig. 1. All the diffraction peaks can be indexed as the tetragonal BiOI (PDF# 10-0445). The absence of any characteristic peaks related to impurities indicates that the sample is only contained pure BiOI.





Fig. S2 shows the  $N_2$  adsorption-desorption isotherms of pure BiOI. It is proved to have a mesoporous structure by the typical type-IV curves with an obvious H3 hysteresis loops, which is due to the slit pores accumulated by flaky particles.



Fig. S3. XPS patterns of BiOI. (a) Survey, (b) Bi 4f, (c) O 1s, (d) I 3d. As seen from Fig. S3a, the surface composition of BiOI is only Bi, O, I, and C elements are observed. The XPS spectra of Bi  $4f_{5/2}$  and Bi  $4f_{7/2}$  are shown in Fig. S3b

which are locate at about 163.72 and 158.41 eV, respectively. In Fig. S3c, the O 1s signal deconvolution of BiOI is 529.79 eV and 531.58 eV. Fig. S3d shows that BiOI has two peaks at 618.75 eV and 630.20 eV, corresponding to I 3d5/2 and I 3d3/2, respectively.



Fig. S4. (a) DRS spectra, (b) Mott-Schottky piots, (c) Photocurrent response spectra and (d) EIS of BiOI

The UV-vis diffuse reflectance spectroscopy (DRS) showed the absorption edge of BiOI (Fig. S4a). As can be seen from the figure, the absorption edge of BiOI is 690 nm. According to the (ahv) 1/2 pair photon energy (hv), the band gap energies (Eg) of BiOI was calculated to be 1.80 eV. Fig. S4b shows a Mote-Schottky (MS) plot of a BiOI, while the flat band potential of the SCE electrode relative to the normal hydrogen electrode (NHE) was -0.12 V. Fig S4c and Fig. S4d shows the photocurrent response and the rate of photogenerated carrier separation of BiOI.