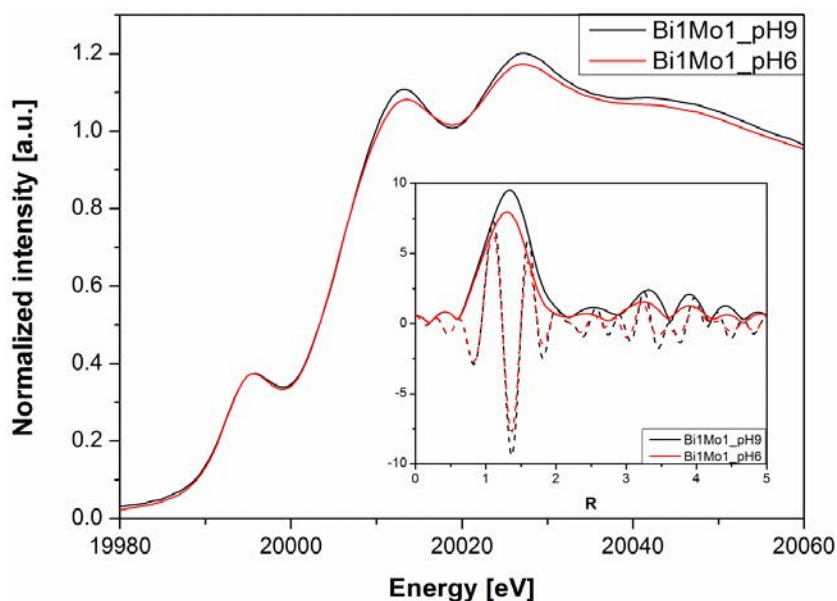
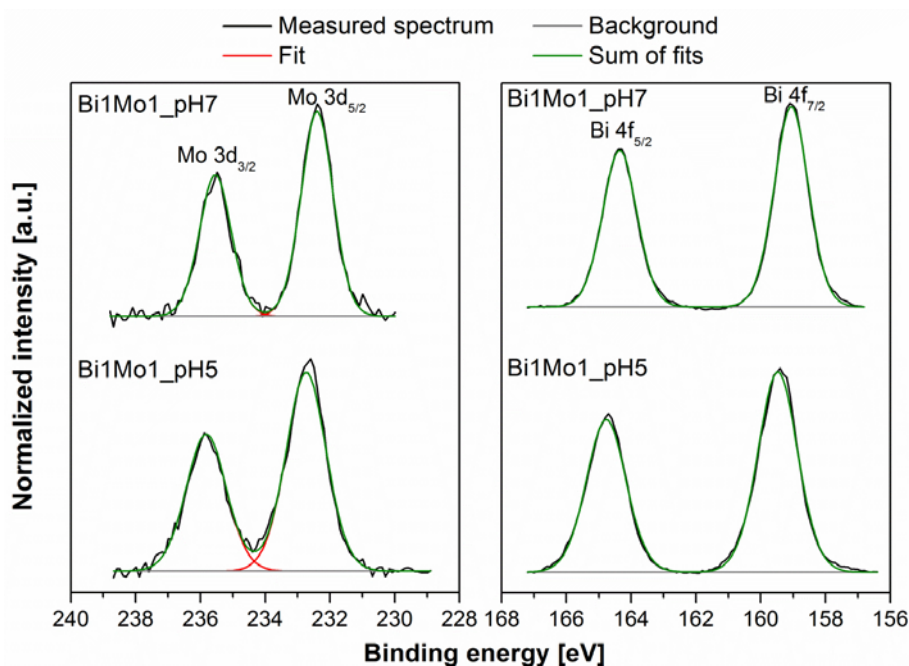


## Supplementary Information

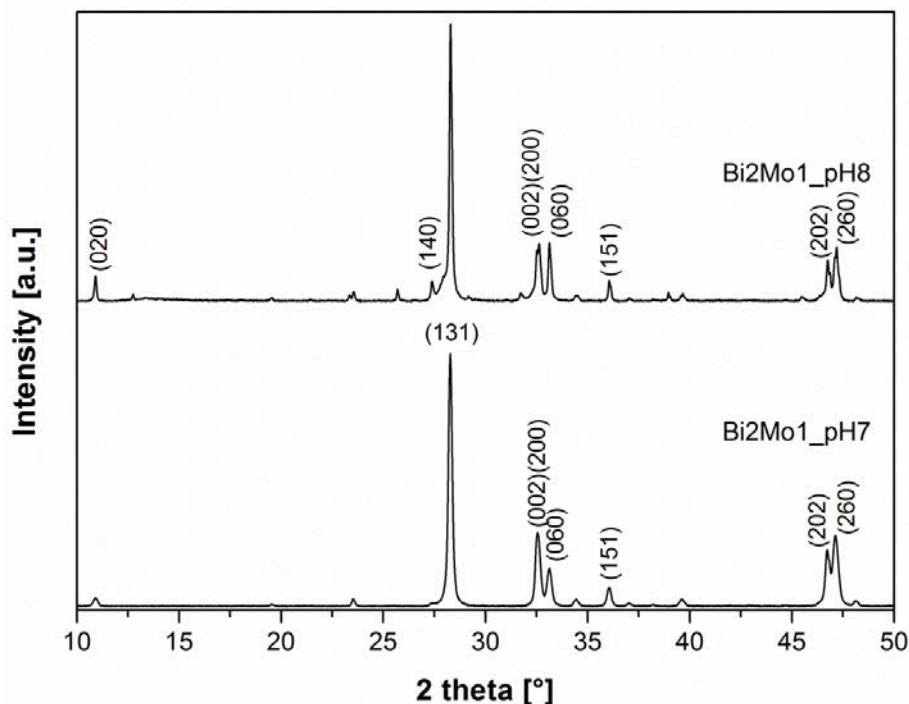
This electronic supporting information gives additional data on X-ray absorption spectroscopy (XANES and EXAFS), X-ray photoelectron spectroscopy (XPS), powder X-ray diffraction (PXRD) patterns and catalytic performance.



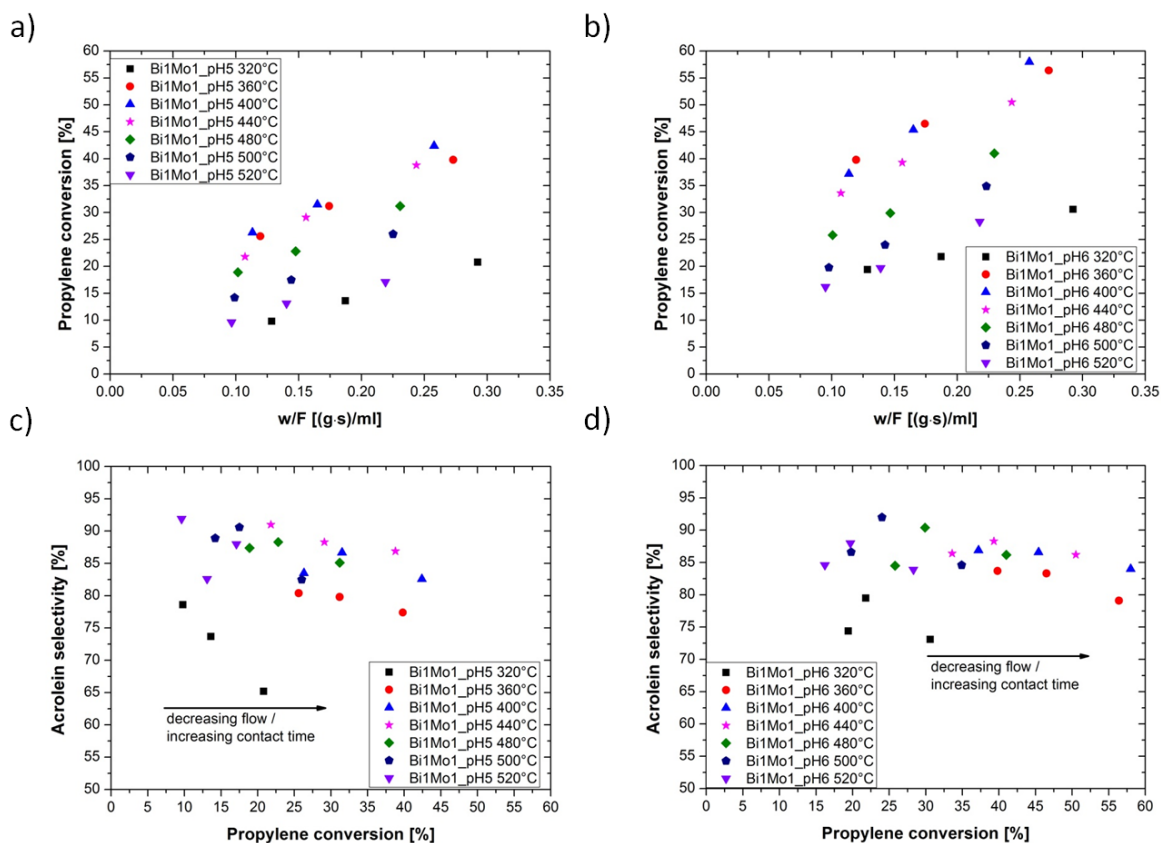
**Figure S1.** XANES and EXAFS of the samples synthesized with Bi/Mo = 1/1 at pH = 6 and pH = 9. Both samples contain only MoO<sub>6</sub> octahedra indicating that the samples consist of pure  $\gamma$ -Bi<sub>2</sub>MoO<sub>6</sub>.



**Figure S2.** XP spectra of Bi1Mo1\_pH7 and Bi1Mo1\_pH5 with the corresponding Voigt fits around the Mo 3d and Bi 4f region. For a better visualization all spectra are normalized to maximum intensity.



**Figure S3.** XRD patterns of  $\gamma$ - $\text{Bi}_2\text{MoO}_6$  prepared with  $\text{Bi}/\text{Mo} = 2/1$  at  $\text{pH} = 7$  and  $8$ .



**Figure S4.** Catalytic performance of the high surface area sample  $\text{Bi}/\text{Mo} = 1/1$  synthesized at  $\text{pH} = 5$  (**a,c**) and the highly active sample synthesized at  $\text{pH} = 6$  (**b,d**) at various temperatures between  $320^\circ\text{C}$  and  $520^\circ\text{C}$  at 50, 80 and 120  $\text{Nml}/\text{min}$  and with  $\text{C}_3\text{H}_6/\text{O}_2/\text{N}_2 = 5/25/70$  and 500 mg of catalyst.

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