Electronic Supplementary Material (ESI) for Physical Chemistry Chemical Physics. This journal is © the Owner Societies 2021

## **Supplementary Information**

## Integrating Trace Amounts of Pd Nanoparticles into Mo<sub>3</sub>N<sub>2</sub> Nanobelts for Improved Hydrogen Evolution Reaction

Saheed Abiola Raheem, <sup>ac</sup> Hanghia Shen, <sup>a</sup> Tiju Thomas <sup>\*b</sup> and Minghui Yang <sup>\*a</sup>

<sup>a</sup> Ningbo Institute of Materials Technology and Engineering, Chinese Academy of Sciences, 1219 Zhongguan West Road, Ningbo 315201, P. R. China. E-mail: <u>myang@nimte.ac.cn</u>

<sup>b</sup> Department of Department of Metallurgical and Materials Engineering, Indian Institute of Technology Madras, Adyar, Chennai 600036, Tamil Nadu, India

<sup>c</sup> University of Chinese Academy of Sciences, Beijing 100049, China



**Figure S1.** (a, b) Low resolution SEM images of  $Mo_3N_2$  nanobelts. (c, d) Low resolution SEM images of 1.5 wt%Pd/Mo<sub>3</sub>N<sub>2</sub> nanobelts.



Figure S2. (a, b) TEM images of  $Mo_3N_2$  and 0.75 wt%Pd/Mo $3_3N_2$  nanobelts. (c) HRTEM image of 0.75 wt%Pd/Mo $_3N_2$ .

Element	Weight%	Atomic%
NK	5.74	29.58
Мо К	93.16	69.79
Pd L	1.09	0.73
Total	99.99	

Table S1. Contents of Pd in catalysts from elemental analyzer

Table S2. Contents of Pd in catalysts from ICP-OES analysis

Sample	Pd (weight %)
$0.5Pd/Mo_3N_2$	0.41
$0.75 Pd/Mo_3N_2$	0.68
$1.5Pd/Mo_3N_2$	1.2



Figure S3. (a) EDX spectra of 0.75 wt%Pd/Mo<sub>3</sub>N<sub>2</sub>. (b) High resolution XPS spectra of O 1s.



**Figure S4.** Cyclic voltammograms with a scan rate of 40, 60, 80, 100 and 120 mV s<sup>-1</sup> in 1 M KOH for  $Mo_3N_2$  (a), 0.5 wt%Pd/Mo<sub>3</sub>N<sub>2</sub> (b), 0.75 wt%Pd/Mo<sub>3</sub>N<sub>2</sub> (c), and 1.5 wt%Pd/Mo<sub>3</sub>N<sub>2</sub> (d).



**Figure S5**. (a) Capacitive current as a function scan rate for  $Mo_3N_2$ , 0.5 wt%Pd/ $Mo_3N_2$ , 0.75 wt%Pd/ $Mo_3N_2$ , and 1.5 wt%Pd/ $Mo_3N_2$  electrocatalysts. (b) LSV curves before and after durability test.



Figure S6. TEM and HRTEM images of 0.75 wt%Pd/Mo<sub>3</sub>N<sub>2</sub> after the stability test.

Catalysts	Pd	Electrolyte	Mass loading	Overpotential	at 10 Tafe	l slope Ref.
	(mg)		(mg cm <sup>-2</sup> )	mA cm <sup>-2</sup> (mV)	(mV dec⁻¹)	
PdCuRu 7.6	0.1 M	КОН	0.2	31	52	1
Pd/G/ZnO/NF	0.29	1 М КОН	-	31	46.5	2
Pd-Pt	17.4	1 М КОН	0.02	71	31	3
$Pd-g-C_3N_4$	14.9	0.5 H <sub>2</sub> SO <sub>4</sub>	0.04	55	35	4
Mn <sub>3</sub> N <sub>2</sub> /PdO	18	0.5 M H <sub>2</sub> SO <sub>4</sub>	0.24	44.6	49.6	5
Pd@CoFe	13.85	1 М КОН	0.27	214	120	6
/NCNT						
PdSn	63.6	$1 \text{ M H}_2\text{SO}_4$	2.75	68	204	7
C-PdPt	4.06	0.5 M H <sub>2</sub> SO <sub>4</sub>	0.26	26	33	8
$0.75Pd/Mo_3N_2$	7.9	0.5 M H <sub>2</sub> SO <sub>4</sub>	0.2	41	88	This work
0.75Pd/Mo <sub>3</sub> N <sub>2</sub>	7.9	1 M KOH	0.2	65	70	This work

Table S3. Comparison of HER performance of  $0.75Pd/Mo_3N_2$  with other Pd-based electrocatalysts in electrolyte.

## Reference

- M. Li, M. Luo, Z. Xia, Y. Yang, Y. Huang, D. Wu, Y. Sun, C. Li, Y. Chao, W. Yang, W. Yang, Y. Yu and S. Guo, J. Mater. Chem. A, 2019, 7, 20151-20157. https://doi.org/10.1039/C9TA06861A
- 2 N. Wang, B. Tao, F. Miao and Y. Zang, RSC Adv., 2019, **9**, 33814-33822. https://doi.org/10.1039/C9RA05335B
- J. Fan, K. Qi, L. Zhang, H. Zhang, S. Yu and X. Cui, ACS Appl. Mater. Interfaces 2017, 9, 21, 18008–18014.

https://doi.org/10.1021/acsami.7b05290

- 4 T. Bhowmik, M. K. Kundu and S. Barman, ACS Catal., 2016, **3**, 1929–1941. https://doi.org/10.1021/acscatal.5b02485
- 5 K. C. Majhi and M. Yadav, J. Alloy. Compd., 2021, **855**, 157511. https://doi.org/10.1016/j.jallcom.2020.157511
- R. Manjunatha, L. Dong, Z. Zhai, J. Wang, Q. Fu, W. Yan and J. Zhang, Green Energy Environ.,
  2020, 2468-0257.

https://doi.org/10.1016/j.gee.2020.12.0055

- 7 A. Kumar and S. Deka, Appl. Catal. A, 2020, **599**, 117575. https://doi.org/10.1016/j.apcata.2020.117575
- 8 B. T. Jebaslinhepzybai, N. Prabu and M. Sasidharan, Int. J. of Hydrogen Energy, 2020, **45**, 11127-11137.

https://doi.org/10.1016/j.ijhydene.2020.02.059