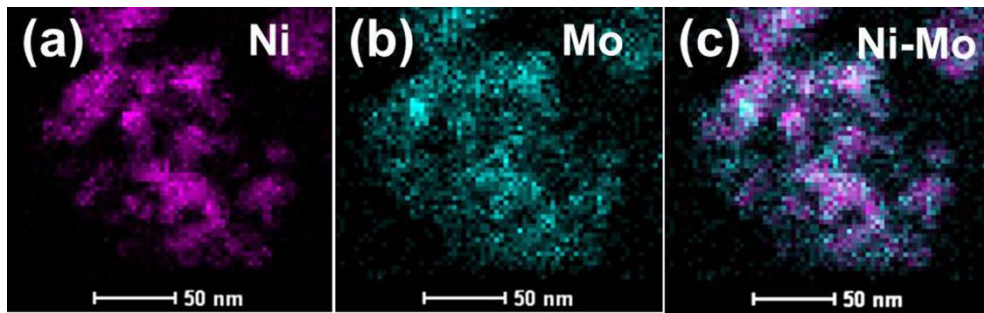


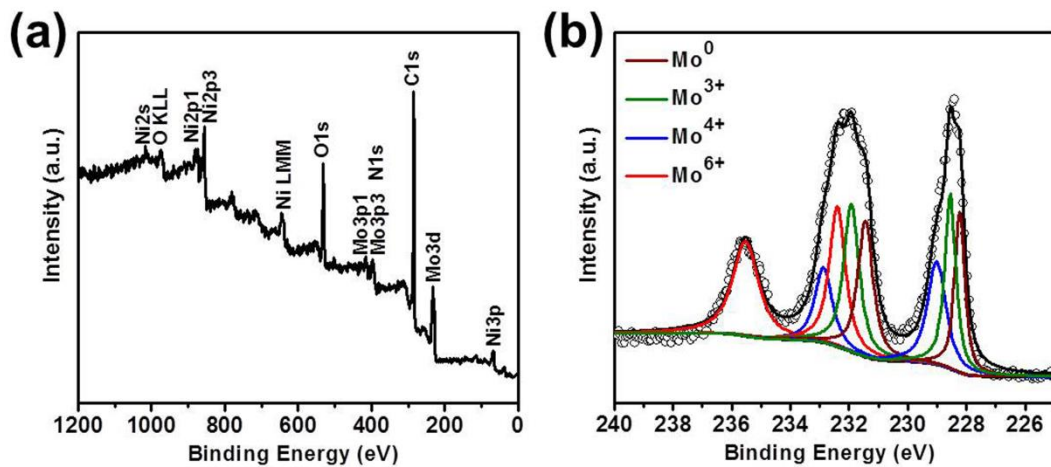


**Table S1.** The preparation parameters for different samples.

Samples	Melamine (mmol)	(NH <sub>4</sub> ) <sub>6</sub> Mo <sub>7</sub> O <sub>24</sub> ·4H <sub>2</sub> O (mmol)	NiCl <sub>2</sub> ·6H <sub>2</sub> O (mmol)
Ni <sub>2.0</sub> Mo <sub>0</sub> @NC	6.0	0	2.0
Ni <sub>0</sub> Mo <sub>0.26</sub> @NC	6.0	0.26	0
Ni <sub>2.0</sub> Mo <sub>0.13</sub> @NC	6.0	0.13	2.0
Ni <sub>2.0</sub> Mo <sub>0.26</sub> @NC	6.0	0.26	2.0
Ni <sub>2.0</sub> Mo <sub>0.39</sub> @NC	6.0	0.39	2.0



**Fig. S1** The elemental mapping images for Ni (a), Mo (b), and their combined image (c).



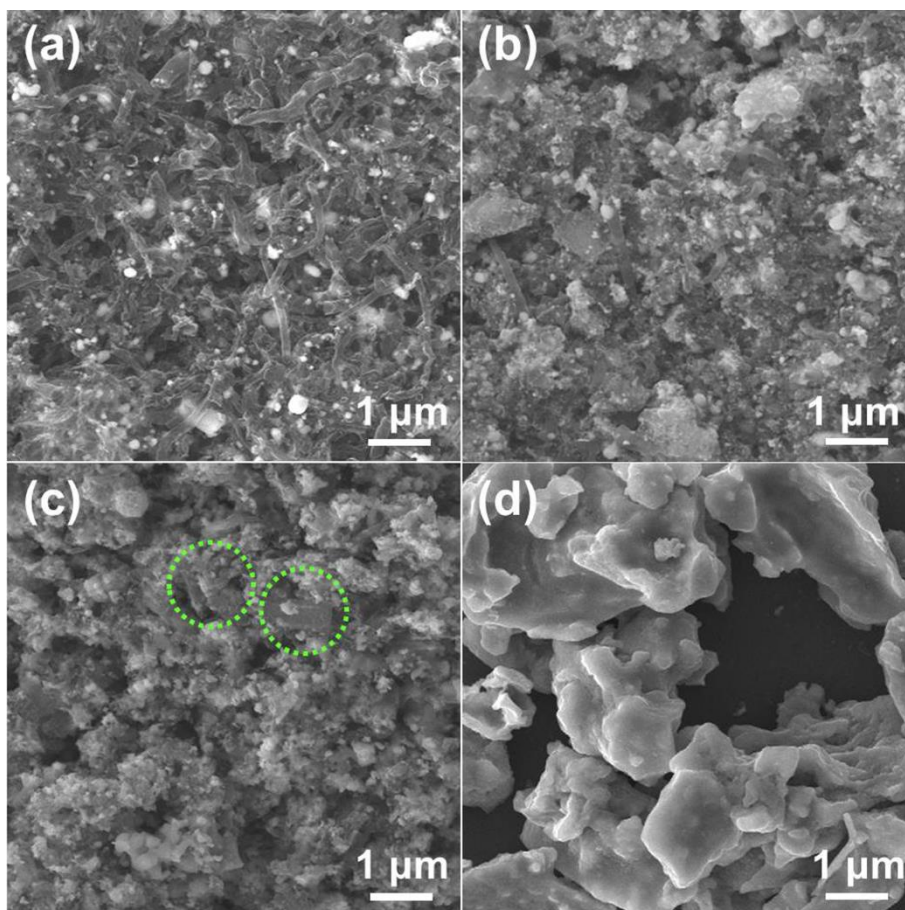
**Fig. S2** (a) XPS survey spectrum and (b) high-resolution XPS spectrum of Mo 3d for sample Ni<sub>2.0</sub>Mo<sub>0.26</sub>@NC.

**Table S2.** Element contents (wt%) of different samples analyzed by XPS.

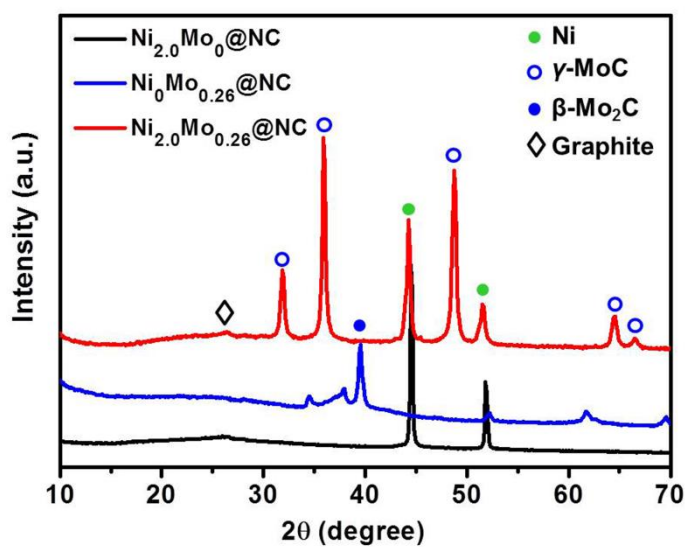
Sample	Mo/XPS	Ni/XPS	C/XPS	N/XPS	O/XPS
Ni <sub>2.0</sub> Mo <sub>0</sub> @NC	0	2.54	88.89	3.42	5.14
Ni <sub>0</sub> Mo <sub>0.26</sub> @NC	13.13	0	67.30	3.93	15.64
Ni <sub>2.0</sub> Mo <sub>0.26</sub> @NC	16.40	5.85	55.14	3.39	19.22
Ni <sub>2.0</sub> Mo <sub>0.13</sub> @NC	11.39	4.61	64.31	4.06	15.63
Ni <sub>2.0</sub> Mo <sub>0.39</sub> @NC	19.42	5.68	54.36	3.24	17.30

**Table S3.** Element contents (wt%) of different samples analyzed by ICP-AES and element analysis.

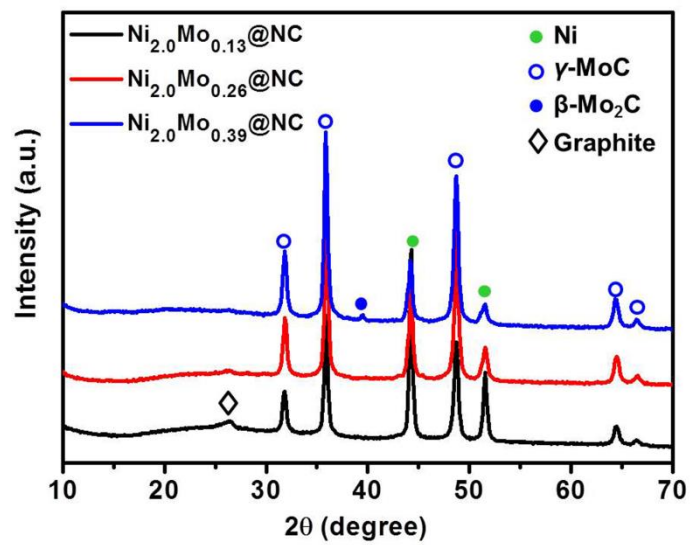
Sample	Mo/ICP	Ni/ICP	C/Element	N/Element	H/Element
Ni <sub>2.0</sub> Mo <sub>0</sub> @NC	0	58.76	36.04	2.01	0.495
Ni <sub>0</sub> Mo <sub>0.26</sub> @NC	71.14	0	11.24	1.80	0.796
Ni <sub>2.0</sub> Mo <sub>0.26</sub> @NC	43.07	30.96	21.79	2.91	0.251
Ni <sub>2.0</sub> Mo <sub>0.13</sub> @NC	34.27	33.53	32.39	3.36	0.391
Ni <sub>2.0</sub> Mo <sub>0.39</sub> @NC	48.54	28.48	18.25	2.97	0.232



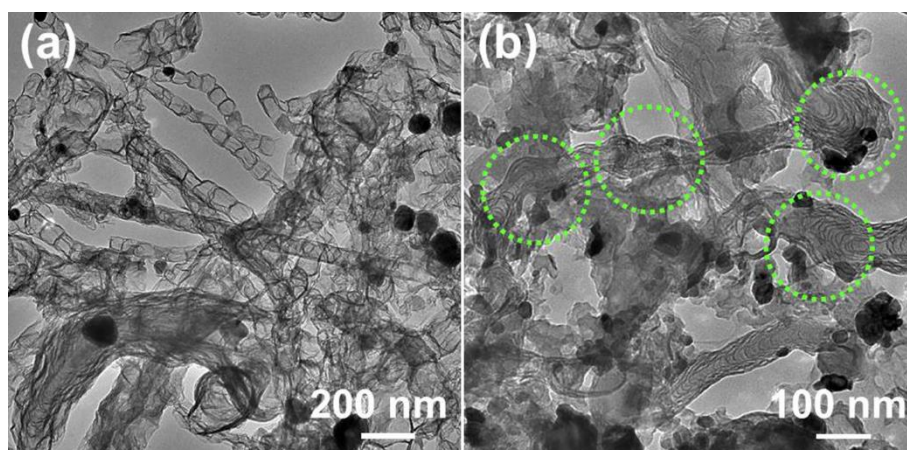
**Fig. S3** SEM images of sample (a)  $\text{Ni}_{2.0}\text{Mo}_0@\text{NC}$ , (b)  $\text{Ni}_{2.0}\text{Mo}_{0.13}@\text{NC}$ , (c)  $\text{Ni}_{2.0}\text{Mo}_{0.39}@\text{NC}$ , and (d)  $\text{Ni}_0\text{Mo}_{0.26}@\text{NC}$ .



**Fig. S4** XRD patterns of sample  $\text{Ni}_{2.0}\text{Mo}_0@\text{NC}$ ,  $\text{Ni}_0\text{Mo}_{0.26}@\text{NC}$  and  $\text{Ni}_{2.0}\text{Mo}_{0.26}@\text{NC}$ .



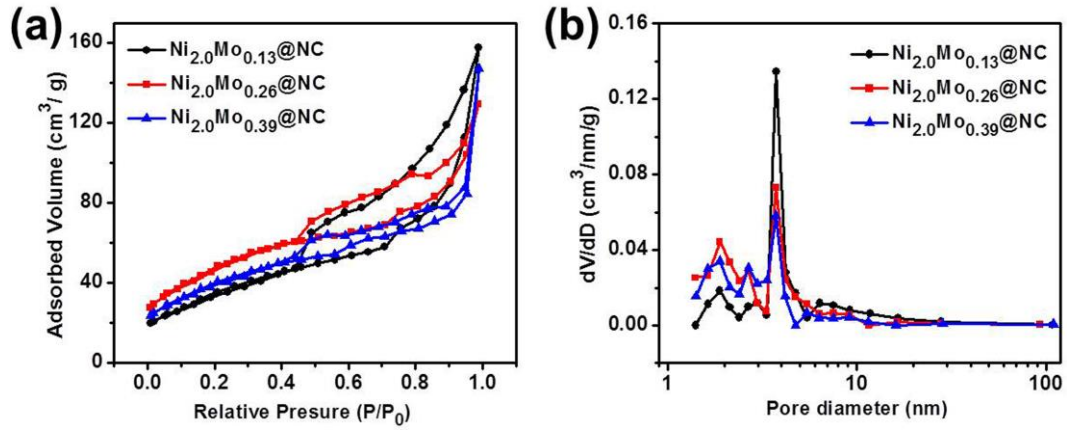
**Fig. S5** XRD patterns of sample  $\text{Ni}_{2.0}\text{Mo}_{0.13}\text{@NC}$ ,  $\text{Ni}_{2.0}\text{Mo}_{0.26}\text{@NC}$  and  $\text{Ni}_{2.0}\text{Mo}_{0.39}\text{@NC}$ .



**Fig. S6** TEM images of sample (a)  $\text{Ni}_{2.0}\text{Mo}_0\text{@NC}$  and (b)  $\text{Ni}_{2.0}\text{Mo}_{0.13}\text{@NC}$ .





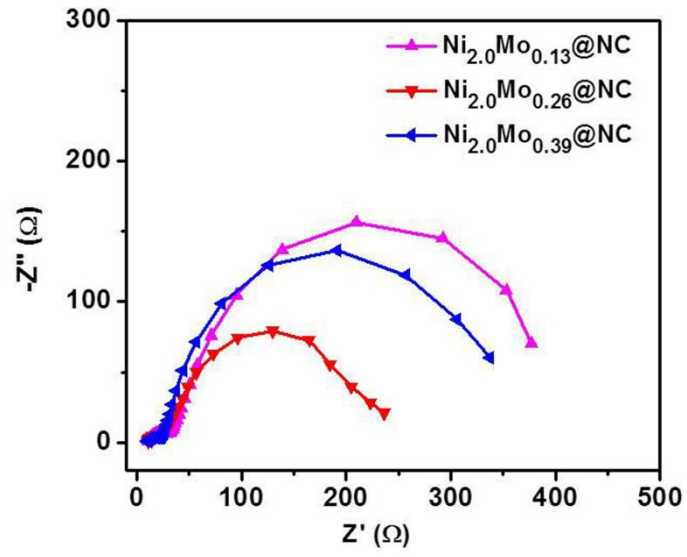


**Fig. S8** (a) Nitrogen adsorption and desorption isotherm and (b) BJH pore distribution of sample  $\text{Ni}_{2.0}\text{Mo}_{0.13}@NC$ ,  $\text{Ni}_{2.0}\text{Mo}_{0.26}@NC$  and  $\text{Ni}_{2.0}\text{Mo}_{0.39}@NC$ .

**Table S6.** The  $\text{N}_2$  sorption data for different samples.

Sample	$S_{\text{BET}}$ ( $\text{m}^2 \text{g}^{-1}$ )	$V_{\text{total}}$ ( $\text{cm}^3 \text{g}^{-1}$ )	$V_{\text{micropore}}$ ( $\text{cm}^3 \text{g}^{-1}$ )	$\text{PD}_{\text{average}}$ (nm)
$\text{Ni}_{2.0}\text{Mo}_{0.13}@NC$	129.7	0.23	0.009	3.75
$\text{Ni}_{2.0}\text{Mo}_{0.26}@NC$	172.0	0.29	0.032	3.74
$\text{Ni}_{2.0}\text{Mo}_{0.39}@NC$	143.2	0.25	0.023	3.74





**Fig. S9** EIS plots of sample  $\text{Ni}_{2.0}\text{Mo}_{0.13}@NC$ ,  $\text{Ni}_{2.0}\text{Mo}_{0.26}@NC$  and  $\text{Ni}_{2.0}\text{Mo}_{0.39}@NC$ .