## **Supplementary Information**

## Small biomolecules sensor based on an innovative MoS<sub>2</sub>-rGO heterostructure modified electrode platform: A binder-free approach

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## MoS<sub>2</sub>-rGO composite



Fig. S1 SEM images of  $MoS_2$ -rGO at different reaction temperatures by keeping the reaction time constant.

## MoS<sub>2</sub>-rGO composite



Fig. S2 SEM images of  $MoS_2$ -rGO at different reaction time by keeping the reaction temperature constant.



**Fig. S3** XRD spectra of MoS<sub>2</sub>-rGO at different reaction temperatures by keeping the reaction time constant.



Fig. S4 XRD spectra of  $MoS_2$ -rGO at different reaction time by keeping the reaction temperature constant.

Fig. S1-S4 clearly shows that the  $MoS_2$ -rGO composites with optimal flower shaped morphology and phase were not obtained until the temperature and reaction time were set to be  $180^{\circ}C/24$  h. Fig. S1 shows the SEM images of  $MoS_2$ -rGO composites at different temperatures (100-160°C) by maintaining the reaction time constant. Fig. S3 demonstrates the corresponding XRD spectra. Fig. S2 presents the SEM images of  $MoS_2$ -rGO composites at different reaction time (4-22 h) by maintaining the temperature constant. Fig. S4 exhibits the corresponding XRD spectra.



**Fig. S5** (a) SEM image of  $MoS_2$ , (b) EDX spectrum of  $MoS_2$ , (c) color mapping image of  $MoS_2$ , and (d-e) elemental color mapping of molybdenum (Mo) and sulfur (S), respectively, (f) color mapping image of  $MoS_2$ -rGO, (g) EDX spectrum of  $MoS_2$ -rGO, and (h-j) elemental color mapping of carbon (C), molybdenum (Mo) and sulfur (S), respectively.



Fig. S6  $\rm N_2$  isotherm and corresponding BJH plot for (a-b)  $\rm MoS_2$  and (c-d)  $\rm MoS_2\mbox{-}rGO$  composite.



Fig. S7 AFM 3D view, surface topography, profile plot and roughness measurement of (a-d)  $MoS_2$ , and (e-h)  $MoS_2$ -rGO.

Sample	Image area (µm <sup>2</sup> )	Root-mean square Roughness (R <sub>q</sub> ) (nm)	Average Roughness (R <sub>a</sub> ) (nm)	R <sub>q</sub> /R <sub>a</sub>	Effective Surface area (µm <sup>2</sup> )	Surface Roughness Parameter (R <sub>s</sub> )
MoS <sub>2</sub>	100	12.5576	8.828	1.4224	100.211	1.002
MoS <sub>2</sub> -rGO	100	78.088	49.5861	1.5748	105.762	1.058



Scheme S1. Schematic of AA, DA and UA sensor based on  $MoS_2$ -rGO composite.



Fig. S8 SEM image of *MoS<sub>2</sub>-rGO/GCE*.



**Fig. S9** CV profiles of *MoS*<sub>2</sub>-*rGO/GCE* in the presence of (a) 300  $\mu$ M AA, (b) 100  $\mu$ M DA, and (c) 200  $\mu$ M UA and (d) the mixture of 300  $\mu$ M AA, 100  $\mu$ M DA and 200  $\mu$ M UA, respectively at a scan rate of 10 mV s<sup>-1</sup>.



**Fig. S10** CV profiles of *MoS*<sub>2</sub>-*rGO/GCE* in a mixture of 300  $\mu$ M AA, 100  $\mu$ M DA and 200  $\mu$ M UA at different scan rates (10-500 mV s<sup>-1</sup>).



Fig. S11 DPV of  $MoS_2$ -rGO/GCE in a mixture of 300  $\mu$ M AA, 100  $\mu$ M DA and 200  $\mu$ M UA.



**Fig. S12** CVs of bare GCE,  $MoS_2/GCE$  and  $MoS_2-rGO/GCE$  in 5 mM [Fe(CN)<sub>6</sub>]<sup>3-/4-</sup> aqueous solution containing 0.1 M KCl.



**Fig. S13** EIS data of bare GCE and *MoS*<sub>2</sub>**-***rGO/GCE* in the absence and presence of analytes (AA, DA and UA), inset shows the equivalent circuit used to fit the EIS data.

Parameter	bare GCE	<i>MoS<sub>2</sub>-rGO/GCE</i> (No Analyte)	<i>MoS<sub>2</sub>-rGO/GCE</i> (Only AA)	<i>MoS2-rGO/GCE</i> (Only DA)	<i>MoS2-rGO/GCE</i> (Only UA)	<i>MoS<sub>2</sub>-rGO/GCE</i> (AA, DA and UA)
$R_{s}\left(\Omega ight)$	5.7 × 10 <sup>-12</sup>	6.0259 × 10 <sup>-12</sup>	7.1128 × 10 <sup>-12</sup>	6.967 × 10 <sup>-12</sup>	4.2768 × 10 <sup>-7</sup>	6.3906 × 10 <sup>-12</sup>
CPE (F)	4.21874 × 10 <sup>-3</sup>	9.647 × 10 <sup>-2</sup>	9.577 × 10 <sup>-2</sup>	9.96503 × 10 <sup>-2</sup>	6.683 × 10 <sup>-5</sup>	9.77553 × 10 <sup>-2</sup>
α	0.7	0.7	0.7	0.7	0.7	0.7
$\mathbf{R}_{\mathrm{F}}\left(\Omega ight)$	$1.01675 \times 10^{5}$	$2.098 \times 10^{3}$	$1.38685 \times 10^{3}$	$1.17566 \times 10^{3}$	$2.16213 \times 10^{3}$	$1.14538 \times 10^{3}$

**Table S2.** Circuit parameters generated after the fitting to the given equivalent circuit.



**Fig. S14** CVs (1<sup>st</sup> and 50<sup>th</sup> cycle) of *MoS*<sub>2</sub>-*rGO/GCE* with 50 continuous scan in 5 mM  $[Fe(CN)_6]^{3-/4-}$  aqueous solution containing 0.1 M KCl.



**Fig. S15** Reproducibility test: CV curves of 4 different  $MoS_2$ -rGO/GCE in the presence of 300  $\mu$ M AA, 100  $\mu$ M DA and 200  $\mu$ M UA at a scan rate of 100 mV s<sup>-1</sup>.