Supporting Information

A robust Mn@FeNi-S/graphene oxide nanocomposite as a high efficiency catalyst for the non-enzymatic electrochemical detection of hydrogen peroxide[†]

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1. Synthesis of GO

GO was synthesized from the graphite (GR) powder through a modified Hummers method. Typically, 1.0 g GR, 1.0 g NaNO₃ and 50 mL conc. H_2SO_4 were added into a 250 mL beaker and stirred for 30 min in an ice bath. Then, 3.0 g of KMnO₄ was added to the mixture and the resultant was transferred to a water bath maintained at 35 °C. The whole reaction mixture was diluted with 150 mL of distilled water. Afterwards, 10 mL of 30% H_2O_2 was slowly added and continued for vigorous stirring up to 60 min. The final product was centrifuged and purified by repeated washing with double distilled water until the pH became neutral. Finally, exfoliated GO was obtained by vacuum drying at 65 °C.



Fig. S1. (a) N₂-sorption of GO and Mn@FeNi-S/GO and (b) corresponding bore size distribution.



Fig. S2 (a) FE-SEM overlay image of the Mn@FeNi-S particles, and EDX elemental mapping of the Mn@FeNi-S particles showing the presence of (b) S, (c) Mn, (d) Fe, and (e) Ni elements.



Fig. S3 (a) FE-SEM overlay image of the Mn@FeNi-S/GO nanocomposite, and EDX elemental mapping of the Mn@FeNi-S/GO nanocomposite showing the presence of (b) S, (c) C, (d) Ni, (e) Fe, (f) Mn, and (g) O elements.



Fig. S4 (a) FE-TEM image, (b) overlay image of the Mn@FeNi-S, (c) Fe, (d) Ni, (e), S, (f) Mn, and (g) O elements.



Fig. S5. (a,b) HR-TEM Images of the Mn@FeNi-S/GO nanocomposite, and the elemental mapping of (a_1) Fe (green dots); (a_2) Ni (red dots); (a_3) Mn (orange dots); (a_4) S (magenta dots); (a_5) C (yellow dots); and, (a_6) O elements (blue dots); TEM-EDS line scan of

Mn@FeNi-S/GO nanocomposite (b_1) overlay image, line scan profile (b_2) , and the corresponding elements lines (b_3-b_9) , respectively.



Fig. S6. (a) CV curves of FeS/GO, NiS/GO, and MnS/GO electrodes in N₂-saturated 0.1 M PB (pH 7.0) containing 25 μ M of H₂O₂ at a scan rate of 50 mV s⁻¹.



Fig. S7. (a) CV curves of Mn@FeNi-S/GO electrode in various concentration of H_2O_2 from 25–125 μ m, (b) corresponding plot of current (μ A) versus concentration of H_2O_2 , (c) effect of catalyst

dosage level on GC electrode toward H_2O_2 sensing, (d) various pH studies of Mn-doped FeNi-S/GO electrode in presence of 25 μ M H_2O_2 .



Fig. S8. (a) Amperometric (i-t) response of FeNi-S/GO nanocomposite in various successive addition of H_2O_2 , (b) the corresponding plot of current response versus time (seconds).

Table 1. Comparison of Mn@FeNi-S/GO modified electrode with other previous reported electrode for H₂O₂

detection

Modified electrode	Material preparation	Linear range	LOD	Sensitivity	Ref.
	method/Buffer; pH)	(μ M)	(nM)	(μΑ μΜ ^{−1}	
				cm ⁻²)	
FeS ₂ /GCE ^a	Wet chemical/PB ^{b} (0.1 M;	0.25–9	250	612	S1
	pH 7.0)				
ERGO ^c /AuNPs/GCE	Electrodeposition/PB (0.1 M;	Upto 100	75	527.8	S2
	pH 6.0)				
NiCo ₂ S ₄ /rGO/GCE	Hydrothermal/NaOH (0.1 M)	25-112.5	190	118.5	S3
NiFe-LDH ^d	Hydrothermal/PB (0.1 M; pH	0.5-840	500	1704	S4
	7.0)				
Fe-MOF ^e /rGO/CPE	Hydrothermal/PB (0.1 M; pH	5.0-945	500	5.17	S5
	7.0)				
Mn ₂ CuO ₄ /GCE	Solvothermal/PB (0.05 M;	0.036-9.3 mM	13	3.107	S6
	pH 7.0)				
AgFe _{Amaranth} /GCE	Stirrer/KOH (0.1 M)	Upto 20 mM	100	1350	S7
HNONS/@rGO/GCE	Ex-situ /PB (0.1 M; pH 7.0)	0.25-13.1 mM	375	222.16	S 8
<i>m</i> SiO ₂ /GCE	Stirrer/AB (0.1 M; pH 6.8)	4-10 mM	300	_	S9
FePc-CP ^g /film	Coupling/PB (0.1 M; pH 7.0)	0.1-1000	17	97.0	S10
FeNi-S/GO/GCE	Heating/PB (0.1 M; pH 7.0)	0.07-123	24.26	2.088	this
					work
Mn@FeNi-S/GO/GCE	Heating/PB (0.1 M; pH 7.0)	0.055-523	8.84	8.929	this
-					work

^{*a*}Glassy carbon electrode. ^{*b*}Phosphate buffer. ^{*c*}Electrochemically reduced graphene oxide. ^{*d*}layered double hydroxides. ^{*f*}Metal organic framework. ^{*e*}Hexagonal nickel oxide nanosheets. ^{*g*}Iron phthalocyanine-conjugated polymer.

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