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Electronic Supporting Information

In-situ developed $NiCo_2O_4$-$Ti_3C_2T_x$ nanohybrid towards non-enzymatic electrochemical detection of glucose and hydrogen peroxide

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Materials and methods

MAX phase with approximately 40 mesh size were purchased from Nano Research Element, India. Lithium fluoride (LiF), ascorbic acid (AA) and hydrogen peroxide (H_2O_2) dopamine hydrochloride (DA) were purchased from Sigma Aldrich, India. Cobalt (II) chloride hexahydrate, nickel (II) chloride hexahydrate, uric acid (UA), cholesterol (Chol), glucose (Glu), tryptophan (L-Tryp), potassium chloride (KCl) and cystine (Cys) were obtained from SRL chemicals, India. Potassium perchlorate ($KClO_3$) was produced from Alfa Aesar, India. Leucine (Leu) was obtained from Sd Fine Chemicals, India and potassium nitrite (KNO_3) was purchased from Avra Chemicals, India. Valine (Val) was bought from Spectrochem, India. All of the acquired compounds were of AR grade and were utilized without additional purification. All sample solutions were prepared using Milli-Q water.

Instrumentation

Bruker D8 advance instrument was used to record the powder X-ray diffraction (PXRD) pattern of the synthesized nanohybrid. Thermo-Fisher FEI QUANTA 250 FEG was used to carry out the Field Emission Scanning Electron Microscopic (FE-SEM) analysis. High-resolution transmission electron microscopic (HRTEM) images were obtained on JEOL JEM 2100 with a LaB6 electron source. PHI5000 Version Probe III was employed to examine the X-ray photoelectron spectroscopic (XPS) information. The CHI-760E electrochemical workstation was utilised for all the electrochemical investigations. A standard three-electrode setup was employed, consisting of a platinum coil functioning as the counter electrode, Ag/AgCl as the reference electrode and glassy carbon as the working electrode.

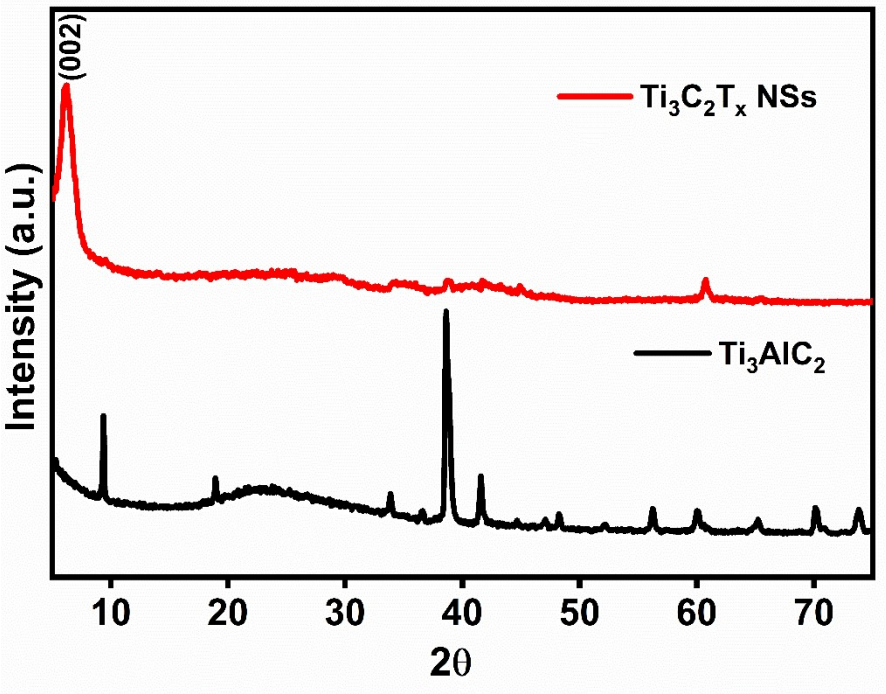

Figure S1. XRD patterns of Ti_3AlC_2 (black) and $Ti_3C_2T_x$ MXene (red).

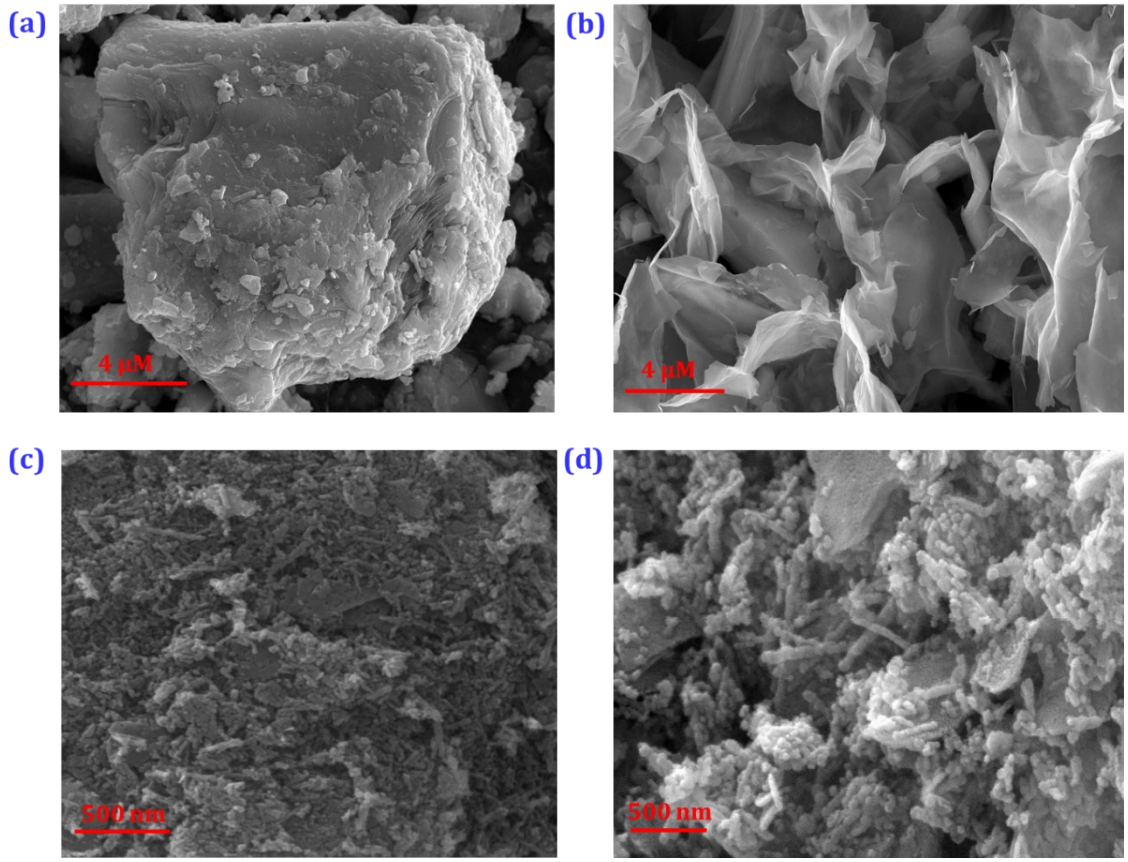

Figure S2. FESEM images of (a) Ti_3AlC_2 (b) $Ti_3C_2T_x$ NSs (c) $NiCo_2O_4$ NPs and (d) $NiCo_2O_4$-$Ti_3C_2T_x$ nanohybrid.

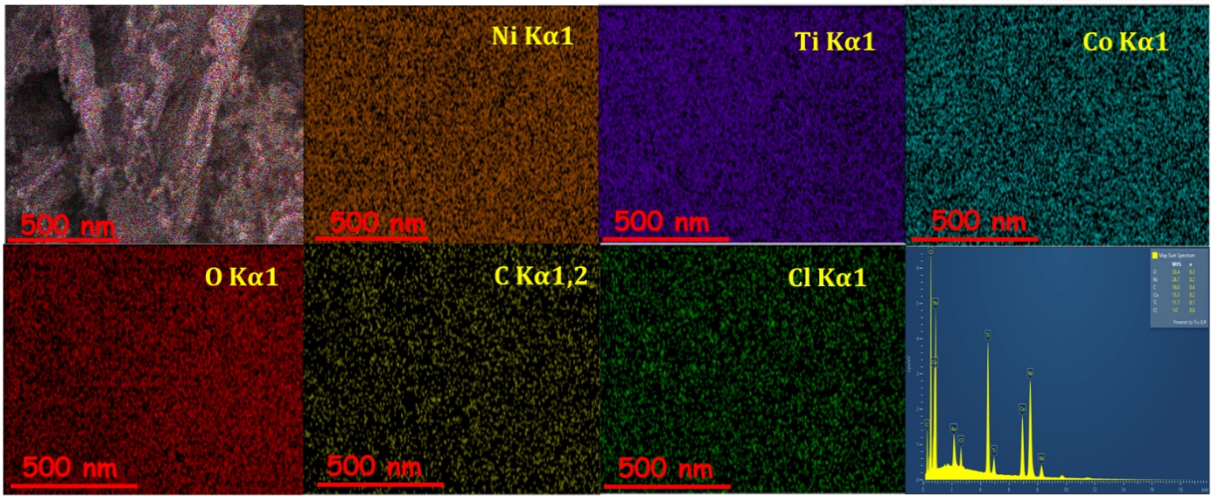

Figure S3. FESEM image and elemental analysis of the $NiCo_2O_4$-$Ti_3C_2T_x$ nanohybrid.

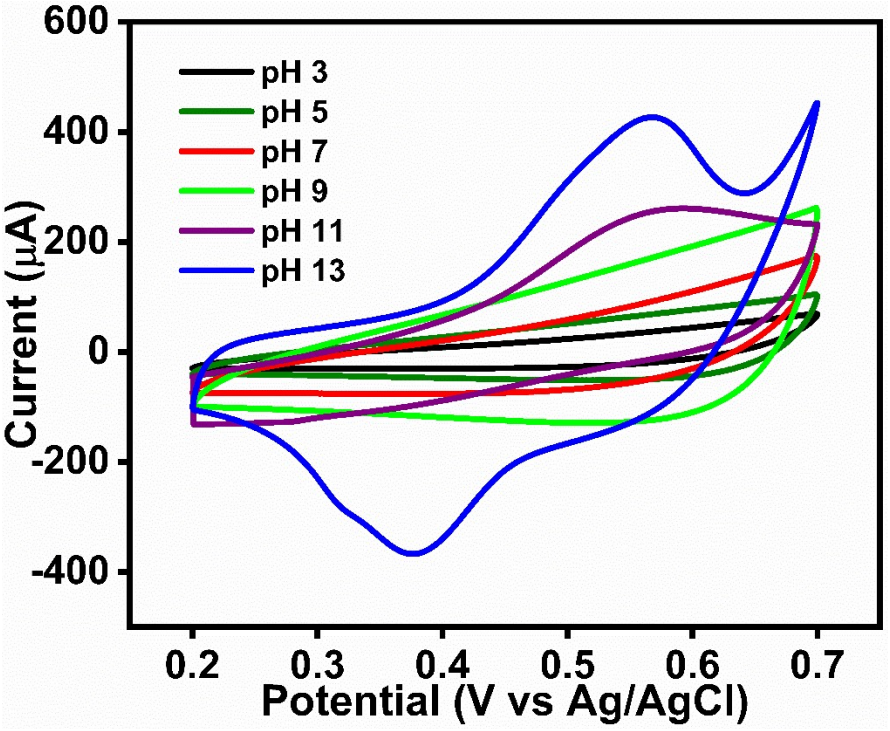

Figure S4. Effect of pH on the electrochemical behaviour of $NiCo_2O_4$-$Ti_3C_2T_x$/GCE.

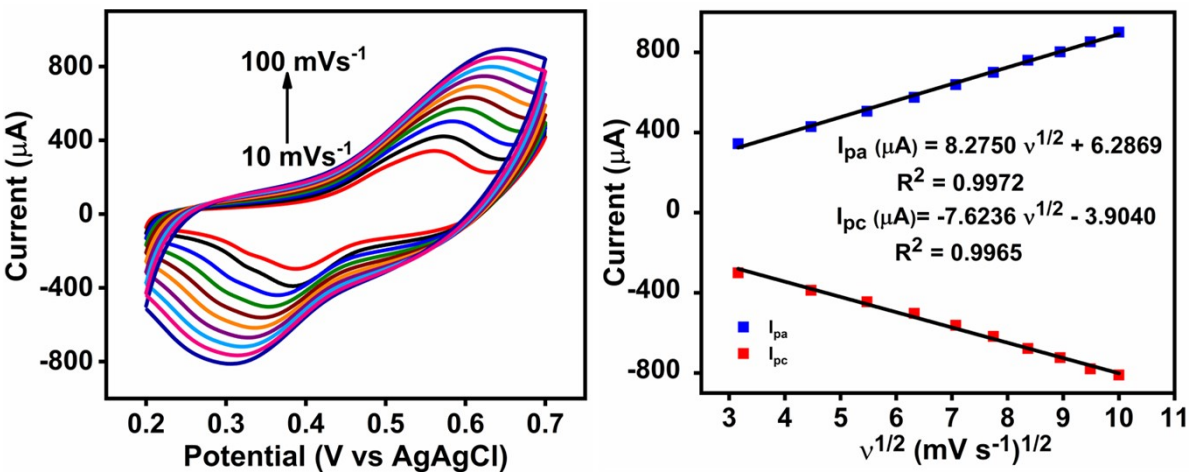

Figure S5. (a) Effect of scan rate and (b) corresponding calibration plot.

Table S1. Real time detection of Glu in serum and urine samples.

Sample	Spiked	Found[a]	Recovery (%)
Serum	100 mM	97.6 mM	97.6
	200 mM	203.1 mM	101.5
Urine	100 mM	98.2 mM	98.2
	200 mM	201.6 mM	100.8

Table S2. Real time detection of H_2O_2 in milk and apple juice samples.

Sample	Spiked	Found[a]	Recovery (%)
Milk	50 mM	48.9 mM	97.8
	100 mM	102.1 mM	102.1
Apple Juice	50 mM	47.8 mM	95.6
	100 mM	105.6 mM	105.6

Electrode material	Analyte	Operating potential (V)	Linear range (μM)	LOD (μM)	Sensitivity	Ref.
MXene/CoNiMn/GCE	Glu	0.5	10 - 900	0.24	-	1
MXene-Cu_2O/GCE	Glu	0.6	0.01 - 3000	2.4	11.061 μA mM^{-1} cm^{-2}	2
Co_3O_4/MXene@CFE	Glu	0.55	0.05 - 7440	0.01	19.3 μA mM^{-1} cm^{-2}	3
Nickel-copper oxide NPs@ 3D-rGO/MWCNT/GCE	Glu	0.6	0.1 - 900	0.04	-	4
$NiCo_2O_4$/RDE	H_2O_2	-0.58	0 - 14000	0.05	392.5 mA mM^{-1} cm^{-2}	5
MX/CS/PB/GCE	H_2O_2	0.0	0.05 - 667	0.004	-	6
CuxO/Ag@FLG/GCE	H_2O_2	-0.65	10 - 100000	2.13	174.5 μA mM^{-1} cm^{-2}	7
Co_3O_4/ATNTs	H_2O_2	-0.6	1.27 - 26.80	6.71	39.53 μA mM^{-1} cm^{-2}	8
$NiCo_2O_4$-$Ti_3C_2T_x$ MXene-GCE	Glu	0.5	30 - 1830	9	101.2 μA μM^{-1} cm^{-2}	This work
	H_2O_2	-0.25	20 - 100 100 -2010	6	107.03 μA μM^{-1} cm^{-2}	This work

Table S3. Comparative performances of $NiCo_2O_4$-$Ti_3C_2T_x$/GCE sensor with recently reported Glu and H_2O_2 sensors.

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