Electronic Supplementary Information

Experimental Section

Materials

Lactose, fructose and glucose (Glu) were purchased from Beijing Chemical Works. Nickel nitrate hexahydrate (Ni(NO₃)₂·6H₂O), cobaltous nitrate (Co(NO₃)₂·6H₂O), sodium hypophosphite (NaH₂PO₂), ammonium fluoride (NH₄F), urea (CO(NH₂)₂), sodium hydroxide acid (AA), uric acid (UA) and dopamine (DA) were purchased from Aladdin Ltd. (Shanghai, China). All reagents were used as received without further purification. Ti mesh was purchased from Phychemi Hong Kong Company Limited. The water use throughout all experiments was purified through a Millipore system.

Preparation of NiCoP/Ti, Ni₂P/Ti, and CoP/Ti

In a typical procedure, Ni(NO₃)₂·6H₂O (1 mmol), Co(NO₃)₂·6H₂O (1 mmol), NH₄F (8 mmol) and CO(NH₂)₂ (10 mmol) were dissolved in 36 mL of distilled water and stirred to form a transparent and homogeneous solution. Prior to utilization, Ti mesh (about 3 cm \times 2 cm) was carefully cleaned with concentrated HCl solution (37 wt.%) in an ultrasound bath for 5 min in order to remove the surface TiO layer, and then deionized water and absolute ethanol were used for 5 min each to ensure the surface of the Ti mesh was well cleaned. The aqueous solution and the Ti mesh were transferred to a 40 mL Teflon-lined stainless-steel autoclave, which was sealed, maintained at 100 °C for 8 h, and then allowed to cool to room temperature naturally. The NiCo precursor was taken out and washed with water thoroughly before dried at 80 °C for 6 h. To prepare NiCoP /Ti, NiCo precursor and 100 mg NaH₂PO₂ were put a porcelain boat with NaH₂PO₂ at the bottom of the porcelain. Subsequently, the samples were heated at 300 °C for 2 h in a flow Ar atmosphere. Ni₂P/Ti and CoP/Ti were converted from corresponding binary precursors.

Preparation of human blood serum

The human blood was put into 45 °C water bath for 10 minutes and then centrifuged at 3000 r/min for 5 minutes. Human blood serum was obtained by collected the

supernate. The disposed serum is kept in - 5 °C to - 20 °C.

Characterizations

Powder X-ray diffraction (XRD) data were acquired on a RigakuD/MAX 2550 diffractometer with Cu K α radiation (λ =1.5418 Å). Scanning electron microscopy (SEM) measurements were performed on a Hitachi S-4800 field emission scanning electron microscope at an accelerating voltage of 20 kV. Transmission electron microscopy (TEM) measurements were made on a Hitachi H-8100 electron microscopy (Hitachi, Tokyo, Japan) with an accelerating voltage of 200 kV. X-ray photoelectron spectroscopy (XPS) measurements were carried out on an ESCALABMK II X-ray photoelectron spectrometer using Mg as the exciting source.

Electrochemical measurements

Electrochemical measurements were performed with a CHI 660E electrochemical analyzer (CH Instruments, Inc., Shanghai) in a conventional three electrode system, using NiCoP/Ti as working electrode, platinum wire as counter electrode and saturated calomel electrode as reference electrode. All tests were carried out at room temperature.



Fig. S1. SEM images for bare Ti mesh.



Fig. S2. EDX spectrum of NiCoP/Ti.



Fig. S3. CVs for Ni₂P/Ti (curve 1), CoP/Ti (curve 2) and NiCoP/Ti (curve 3) in 0.1 M NaOH with 1 mM Glu (scan rate: 30 mV/s).



Fig. S4. Amperometric responses of (a) CoP/Ti and (b) Ni₂P/Ti in 0.1 M NaOH to the consecutive addition of Glu.

Catalysts	Sensitivity (µA	Linear	Detection	Ref.
NiO nanowalls	2300	0.0002-1	0.2	1
600-NiO/SiC	2037	0.004-7.5	0.32	2
NiO-MWCNT/CPE	6527	0.001-14	19	3
NiO nanoflake	8500	0.01-0.8	1.2	4
c-Ni(OH) ₂ HR	1569	0.002-3.8	0.6	5
PI/CNT-Ni(OH) ₂	2071.5	0.001-0.8	0.36	6
rGO/Ni(OH) ₂	11400	0.02-30	15	7
Ni(OH) ₂ /3DGF	2650	0.001-1.17	0.34	8
Ni(OH)2@oPPyNW	1049	0.001-3.86	0.3	9
Ni ₂ P NA/CC	7792	0.001-3.0	0.18	10
NiO-HMSs/GCE	2390	0.00167-0.42	0.53	11
NiO-MWCNT/CPE	931.4	0.001-0.5	38	12
NiO-MWCNT/CPE	1552	0.001-0.5	58	12
NiO-MWCNT/CPE	3818.5	0.001-0.5	24.5	12
NiO-MWCNT/CPE	6527	0.001-14	19	12
CoFe-LDH	25.6	0.1-10	1.40	13
CoP /Ti	4122.6	0.001-6.0	0.70	This work
Ni ₂ P /Ti	2289.5	0.001-6.0	0.40	This work
NiCoP /Ti	14586	0.001-6.0	0.13	This work

 Table S1. Performance comparison of non-enzymatic Glu electrochemical sensors

 using different catalyst materials.



Fig. S5. Thevariation in the response current of NiCoP/Ti toward 1 mM Glu in 0.1 M NaOH for 25 days (scan rate: 30 mV/s).

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