Supporting Information of:

Ultra-fast and higher sensitive enzyme-free glucose biosensing on nickel-nickel oxide core-shell electrode

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1. Electrochemical and Instrumental Characterization

All electrochemical experiments were performed using a three-electrode potentiostat (CHI 700C electrochemical workstation, CH instrument, USA). A standard three-electrode setup was used with a GCE working electrode (geometric area: 0.0707 cm^2), a Pt wire and an Ag/AgCl were used as counter electrode and reference electrode, respectively. Electrochemical impedance spectroscopy (EIS) was performed with a Versa State 3, manufactured by Metek, USA. All glucose sensing experiments were performed in an Ar purged 0.1 M NaOH in deionized water solution at room temperature (RT). A commercial glucose meter, ACCU-CHEK Performa, was used for glucose determination. The surface morphology of all NiNiO core-shell was characterized by a JSM-7500F JEOL for field emission scanning electron microscope (FESEM) and energy-dispersive X-ray spectroscopy (EDX). Transmission electron microscopy (TEM) images and were obtained by TECNAI model FI-20 (FEI, Netherland). X-ray photoelectron spectroscopic (XPS) spectrum was gained using a MultiLab 2000 with a 14.9 keV Al K X-ray

source. X-ray diffraction (XRD) spectra were carried out on a Rigaku D/max-2500, using filtered Cu Kα radiation.

2. Treatment of human urine

Human urine was obtained from a healthy volunteer. 50 mL was centrifuged at 3287 g (7000 rpm) for 10 min at room temperature (~25 °C). The urine sample was analyzed immediately and was stored in refrigerator at low temperature until analysis.

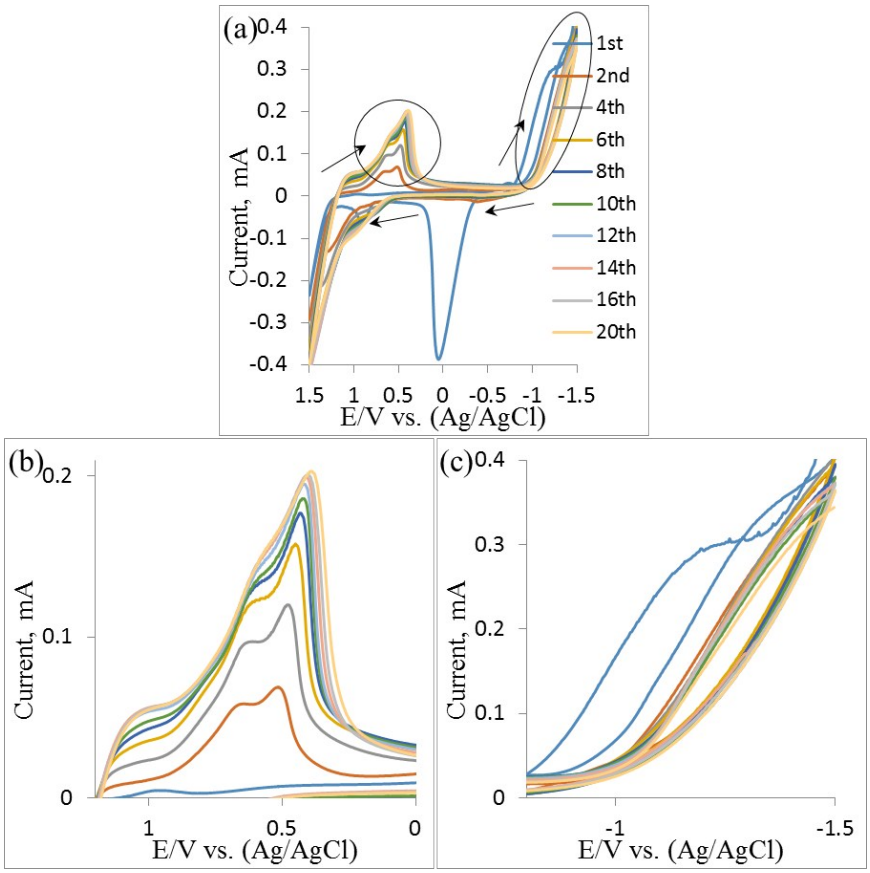

Figure S1: CVs of NiNiO electrodeposition onto GCE in Ar-pursed 2.6 mg ml^{-1} $NiCl_2.6H_2O$/10 mM HCl solution for 20 cycles at 50 mV s^{-1} scan rate (a); enlarged CVs at low overpotential (b) and high overpotential (c).

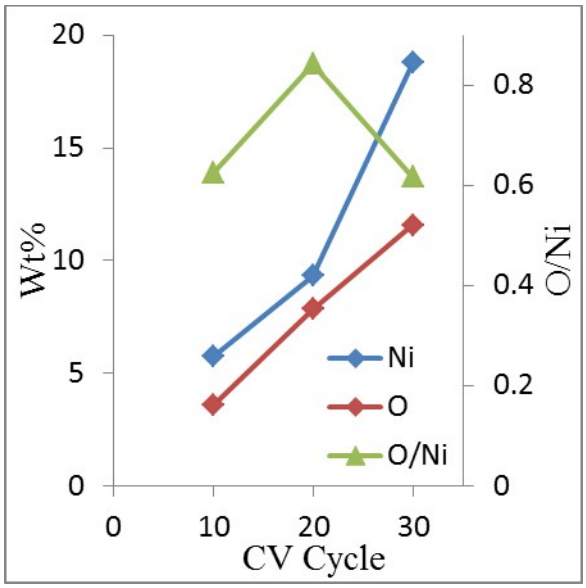

Figure S2: The plot of Ni and O elements with their ratio vs. CV cycles for 10, 20 and 30 CV cycles deposited NiNiO core-shell based on numerical analysis using EDX.

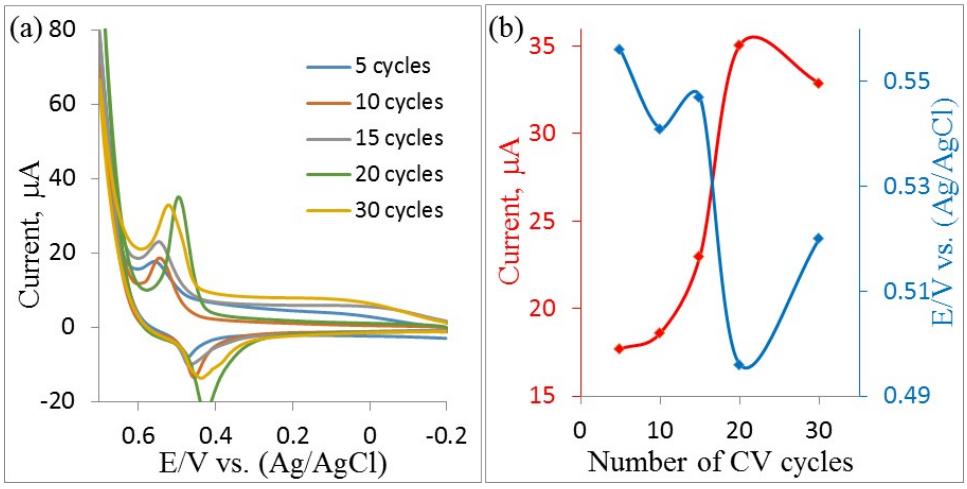

Figure S3: CVs at 50 mV s^{-1} scan rate in Ar-saturated 0.1 M NaOH on 5, 10, 15, 20, and 30 CV cycles deposited NiNiO core-shell-modified GCEs at 50 mV s^{-1} scan rate (a) and the plots of I_{pa} and E_{pa} as the function of CV cycles (b).

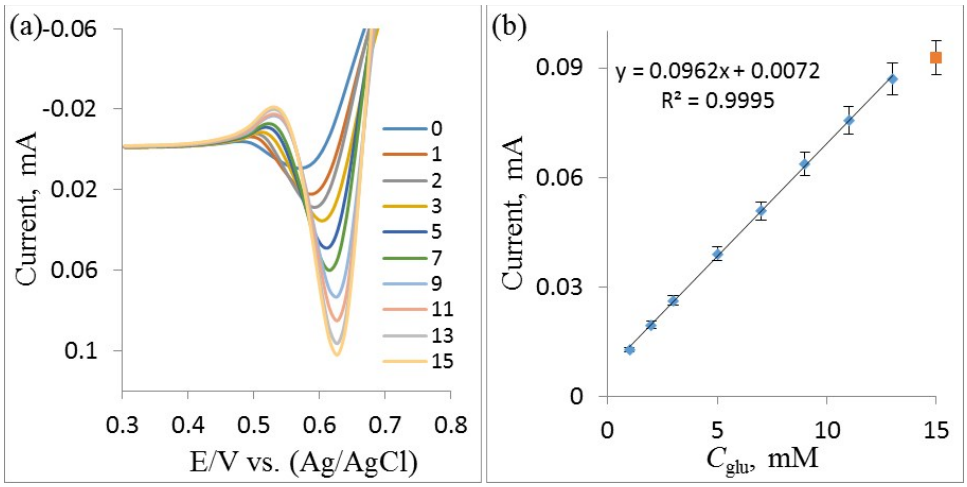

Figure S4: DPVs were recorded in Ar-saturated 0.1 M NaOH on NiNiO/GCE with sequential addition of glucose (a), plot of peak current vs. C_{Glu} with 5% error bar (b).

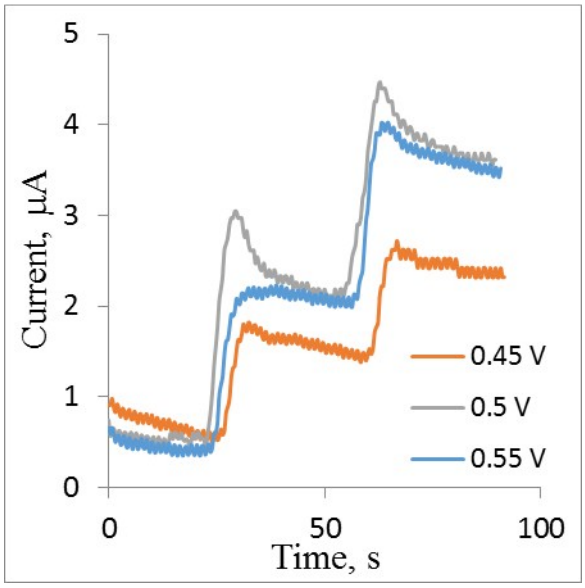

Figure S5: Amperometric response on NiNiO/GCE upon addition of 50 µM glucose in Ar-saturated 0.1 M NaOH at different applied potentials for applied potential optimization.

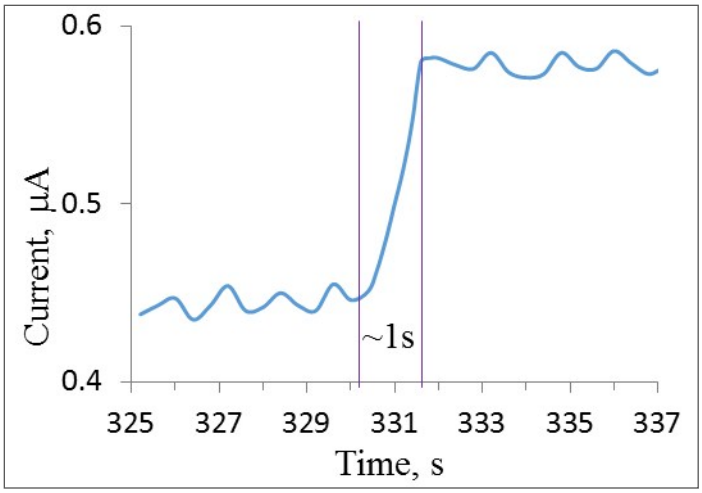

Figure S6: Enlarged CA response on NiNiO/GCE upon addition of glucose in Ar-saturated 0.1 M NaOH at an applied potential of 0.5 V.

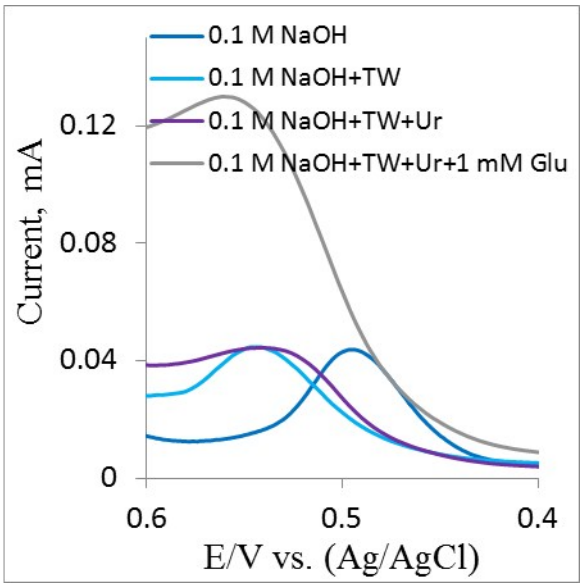

Figure S7: Enlarged CVs on NiNiO/GCE at 50 mV s^{-1} scan rate in Ar-saturated 0.1 M NaOH with subsequent addition of 200 µL TW, 200 µL Ur and 1 mM glucose.

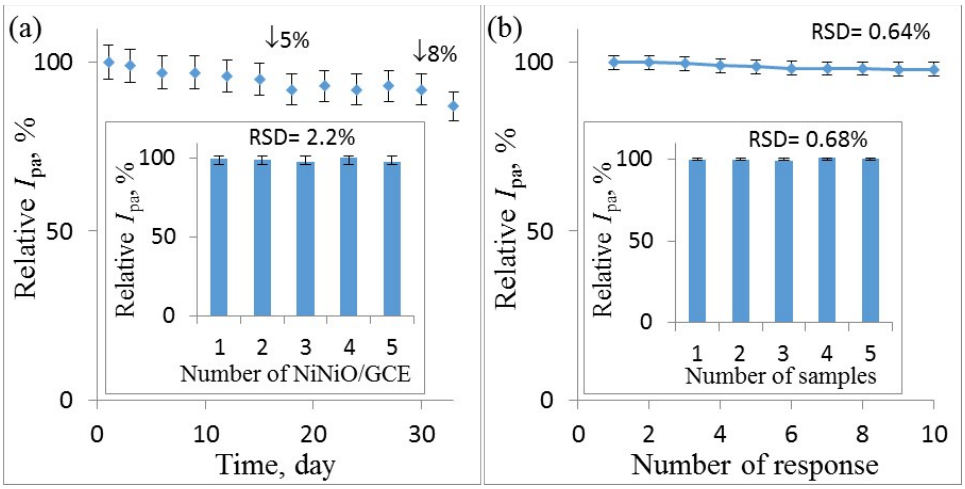

Figure S8: Long term stability evaluation of a single NiNiO core-shell-modified GCE over 33 days with 5% error bar (a), reproducibility test on five different NiNiO/GCEs for the detection of 1 mM glucose with 2.2% RSD (inset), the repeatability tested on a single NiNiO/GCE with 0.64 % RSD (b) and in five different glucose sample solutions with 0.68 % RSD (inset); all tests were evaluated by CV technique in Ar-saturated 0.1 M NaOH.

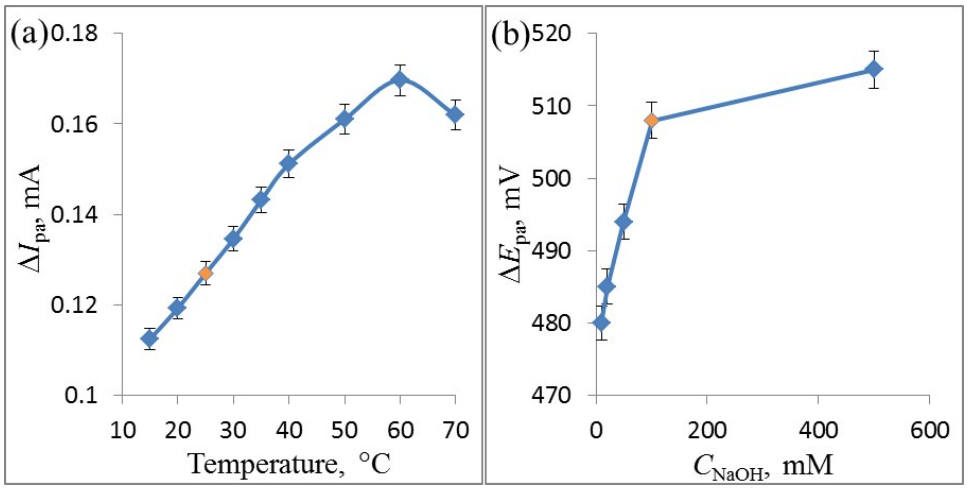

Figure S9: The temperature (a) and NaOH concentration effect (b) on the NiNiO core-shell-modified GCE in Ar-saturated 1 mM glucose containing 0.1 M (a) and 0.01-0.5 M (b) NaOH solution.

Table S1: Glucose detection in urine by a commercial glucose sensor and NiNiO core-shell-modified GCE biosensor method.

Urine Samples	Added glucose (mM)	Glucometer (mM)	Our sensor (mM)	Recovery (%)
1	1.00	1.01	1.02	102
2	3.00	2.98	2.99	99.7
3	5.00	4.98	4.97	99.4