Supplementary Information (SI) for Analyst.
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A Portable Electrochemical Platform for Biorecognition-Free Detection of Ochratoxin A and Ascorbic Acid for Food Safety and Quality Monitoring

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Supporting Information

Supplementary Video 1: Smartphone enabled electrochemical OTA sensing using TeO_2-NiO modified paper electrode.

Structural Characterisation of Synthesised TeO_2-NiO Conjugate

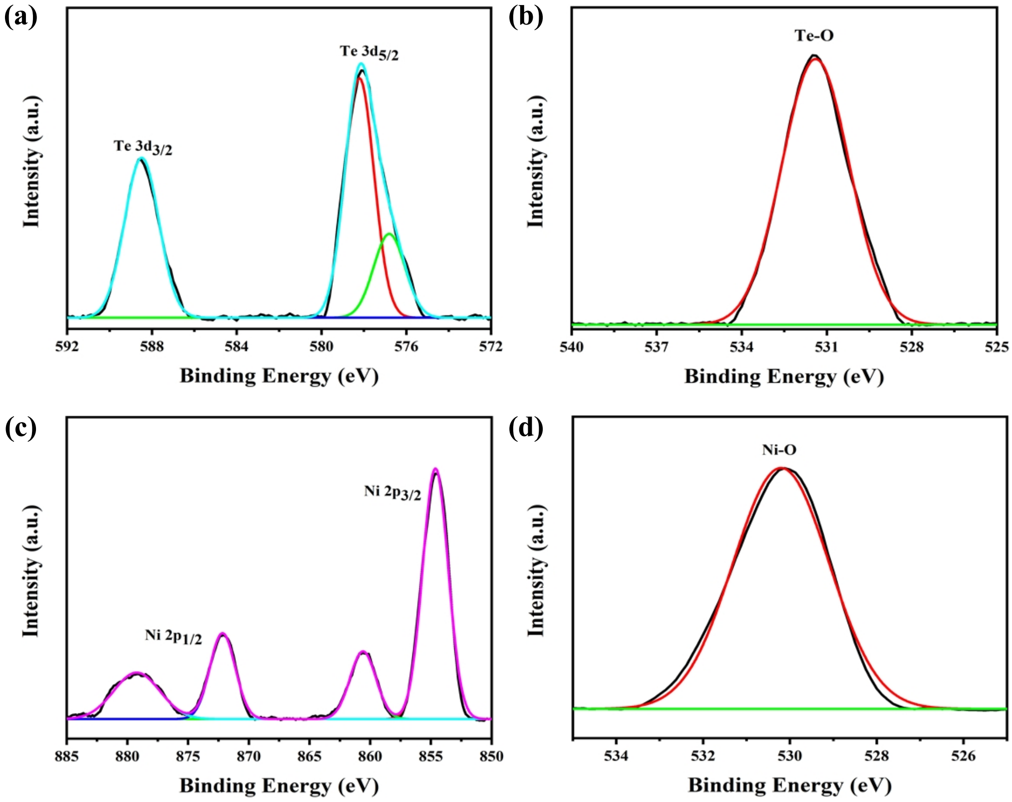

Figure S1: Fitted high-resolution XPS (HR-XPS) spectrum of (a) Te_{3d}, (b) O_{1s} collected from TeO_2, and (c) the empirical fitted spectra of Ni_{2p}, (d) O_{1s} collected from NiO.

Electrochemical behaviour of the modified sensor surface:

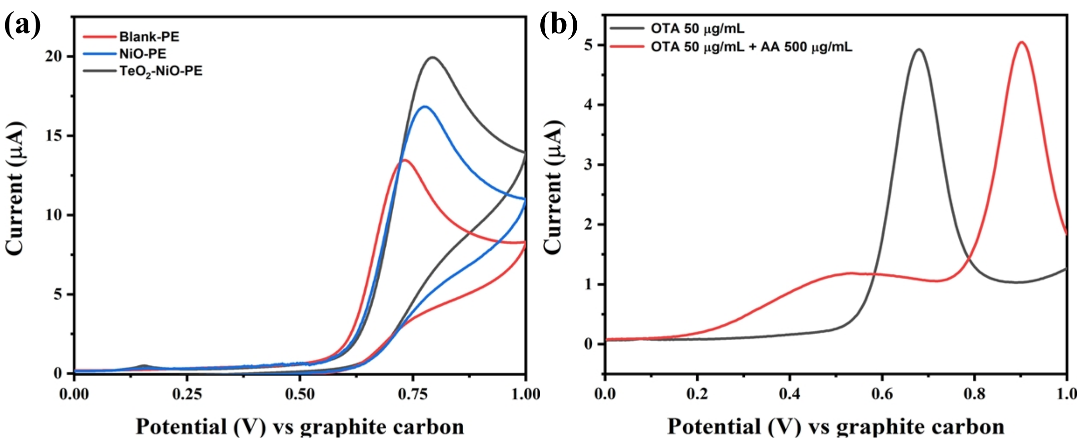

Figure S2: (a) Cyclic voltammetric response of TeO_2-NiO modified paper electrode for ochratoxin-A sensing. The combined effect resulted in an increase in the signal response by approximately 35 %. (b) The differential pulse voltammetric (DPV) response for OTA sensing in the absence (i) and presence (ii) of ascorbic acid (AA) is presented. As observed in the voltammogram, the presence of AA results in a shift of the major peak potential; however, the anodic current remains unchanged.

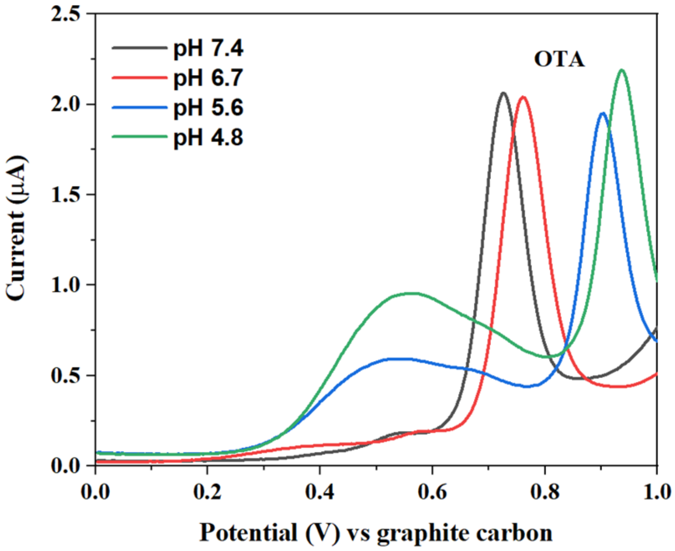

Figure S3: Effect of buffer pH on the electrochemical oxidation of OTA. With the increase in proton concentration (decreasing pH), there is a systemic rightward shift in the DPV peak, where the peak current remains nearly unchanged.

The selection of an appropriate material for the simultaneous detection of multiple analytes presents a significant challenge.[1] The electrocatalytic activity of the modified electrode surface was evaluated with two major criteria: the formation of two distinct oxidation peaks that represent ochratoxin A (OTA) and ascorbic acid (AA), and an enhancement in the oxidation current response. The oxidation peak related to the OTA was carefully monitored to ensure that it was prominent, as AA is relatively simple to detect. The electrochemical mechanism of OTA oxidation was further analysed through a scan-rate study to validate insights. The scan ranges from 10 mV/s to 100 mV/s in 1X PBS (pH 7.4). As the scan rate increased, a thinner diffusion layer formed at the electrode-electrolyte interface, facilitating greater charge transfer and yielding higher current (Figure S3a).[2] A linear relationship between current (I) and scan rate (ϑ) is described in equation (i):

$$log_{I} = \log a + b\log \vartheta \tag{i}$$

with a slope value of 0.51, suggesting a diffusion-controlled oxidation process on the TeO_2-NiO-PE surface (Figure S3b).[2] The solid-state redox transition between Ni^{2+} and Ni^{3+} is facilitated by the incorporation and removal of hydroxyl ions within the oxide lattice.[3] This process maintains a permeable electrode structure, thereby enhancing charge transport from the electrolyte solution to the TeO_2-NiO-modified electrode surface. The Nicholson-Shain equation[4,5] (equation ii) was used to determine the number of electrons transferred during the OTA oxidation process:

$$D = {}^{I^2RT}\Big/_{\left(0.4463\, n\, C_oAF\right)^2 nF\vartheta} \tag{ii}$$

where C_o is the initial concentration and n is the number of electron transfers. The calculated value of electron transfer, 1.8, closely approximates 2, aligning with the expected two-electron transfer mechanism.

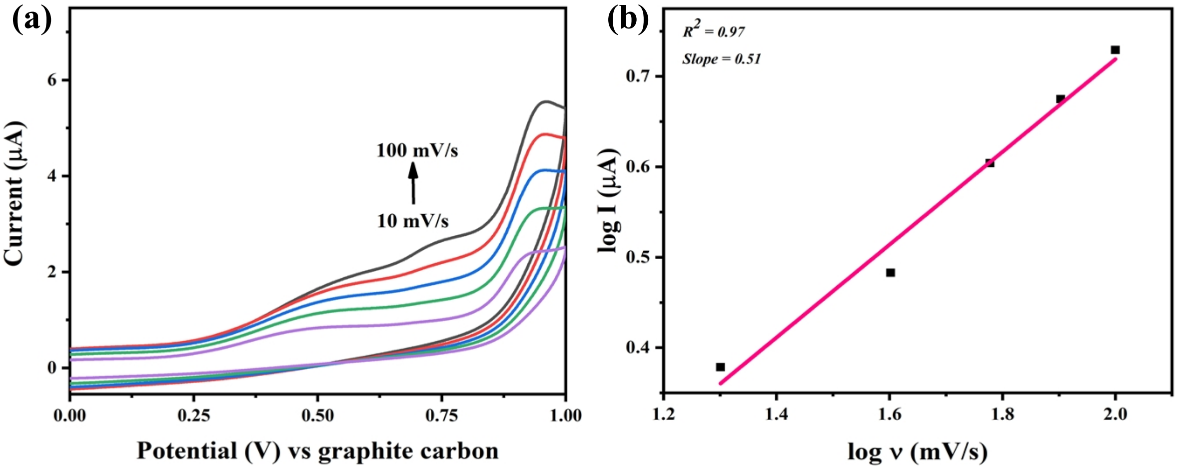

Figure S4: (a) Cyclic voltammetric response of TeO_2-NiO modified paper electrode at different scan rates, (b) Corresponding log I vs log ϑ plot

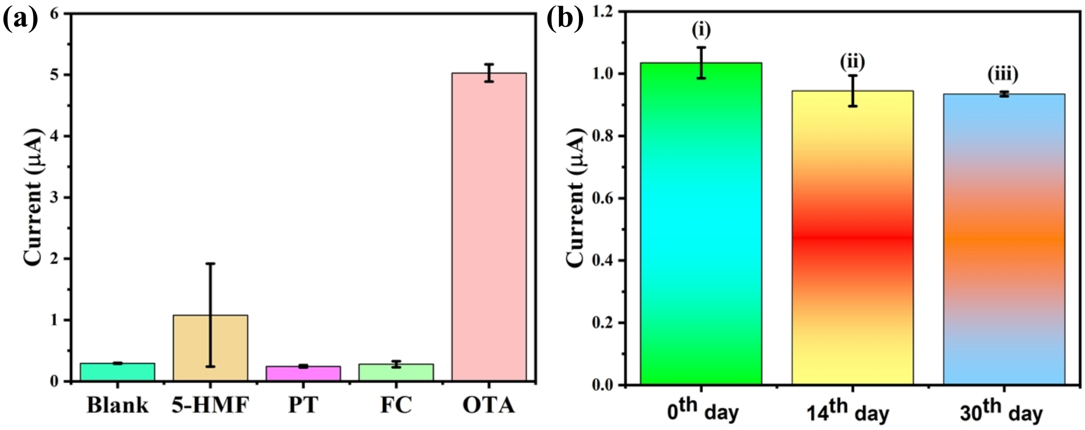

Figure S5: (a) Bar plot for the Selectivity test of Ochratoxin-A sensing with potential interfering molecules, (b) Bar plot for stability analysis of TeO_2-NiO modified paper electrode towards OTA sensing. The voltametric response was recorded from the 0th day (i) to the 14th day (ii) to the 30th day (iii). A signal retention of 98.4 % was recorded from the 0th day to the 30th day.

References:

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