Supporting information

Electrochemical Detection of H2O2 using Bi2O3/Bi2O2Se Nanocomposites

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1. Characterization of the materials

Powder X-Ray Diffraction (XRD) data was collected on XRDynamic 500 diffractometer (Antor Paar). The instrument was equipped with a copper source, operated at a voltage of 45 keV and a power of 40 kW. UV-Visible-Near Infrared Spectra (UV-Vis-NIR) were collected on a Shimadzu UV-Visible-NIR spectrometer equipped with a Harrick Praying Mantis Diffuse Reflection Accessory. Spectra were referenced to a background of Potassium Bromide. FEI XL30 SEM-FEG Scanning Electron Microscope was used to examine the morphology and elemental mapping via Energy Dispersive X-ray (EDX) analysis. XPS experiments were performed using a Physical Electronics VersaProbe III instrument equipped with a monochromatic Al kα X-ray source (hν = 1,486.6 eV) and a concentric hemispherical analyzer. Charge neutralization was performed using both low energy electrons (<5 eV) and argon ions. The binding energy axis was calibrated using sputter cleaned Cu (Cu 2p3/2 = 932.62 eV, Cu 3p3/2 = 75.1 eV) and Au foils (Au 4f7/2 = 83.96 eV). The Bi-compounds were charge referenced to Bi3+ at 159.0 eV in the Bi 4f7/2 spectra. Measurements were made at a takeoff angle of 45° with respect to the sample surface plane. This resulted in a typical sampling depth of 3-6 nm (95% of the signal originated from this depth or shallower). Quantification was done using instrumental relative sensitivity factors (RSFs) that account for the X-ray cross section and inelastic mean free path of the electrons. On homogeneous samples major elements (>5 atom%) tend to have standard deviations of <3% while minor elements can be significantly higher. The analysis size was ~200 µm in diameter. All electrochemical characterization were performed on Waverider 200 Bipotentiostat (Pine Research).



**Figure S1.** CV scans of Bi2O2Se with synthesis time of (a) 10 min (b) 3 hours (c) 6 hours (d) 18 hours (e) 3 days and (f) 7 days.



**Figure S2.** EDX analysis of BOSe-6 h sample.

**Table S1.** Elemental analyses of BOSe-6 h sample from EDX.

|  |  |  |  |  |  |  |  |
| --- | --- | --- | --- | --- | --- | --- | --- |
| **Map Sum Spectrum** | | | | | | | |
| Element | Signal Type | Line | Wt% | Wt% Sigma | Atomic % | Standard Name | Factory Standard |
| C | EDS | K series | 23.28 | 0.08 | 70.21 | C Vit | Yes |
| O | EDS | K series | 6.22 | 0.04 | 14.08 | SiO2 | Yes |
| Se | EDS | L series | 12.20 | 0.05 | 5.60 | Se | Yes |
| Bi | EDS | M series | 58.30 | 0.10 | 10.11 | Bi | Yes |
| Total |  |  | 100.00 |  | 100.00 |  |  |

Ratio of Bi: O: Se = 4:5:2



**Figure S3.** EDX analysis of BOSe-7 days sample.

**Table S2.** Elemental analyses of BOSe-7 days sample from EDX.

|  |  |  |  |  |  |  |  |
| --- | --- | --- | --- | --- | --- | --- | --- |
| **Map Sum Spectrum** | | | | | | | |
| Element | Signal Type | Line | Wt% | Wt% Sigma | Atomic % | Standard Name | Factory Standard |
| C | EDS | K series | 10.01 | 0.09 | 49.04 | C Vit | Yes |
| O | EDS | K series | 5.27 | 0.05 | 19.39 | SiO2 | Yes |
| Al | EDS | K series | 1.24 | 0.04 | 2.71 | Al2O3 | Yes |
| Se | EDS | L series | 11.53 | 0.08 | 8.60 | Se | Yes |
| Bi | EDS | M series | 71.94 | 0.12 | 20.26 | Bi | Yes |
| Total |  |  | 100.00 |  | 100.00 |  |  |

*\*The presence of Al in spectrum could be from the stub holder, or from error estimation due to peak overlap with Se.*

Ratio of Bi: O: Se = 2:2:1



**Figure S4.** XPS analysis of BOSe-6 h sample.

**Table S3.** Elemental analysis of BOSe-6 h from XPS.

|  |  |
| --- | --- |
| **Element** | **Atom%** |
| Bi | 33.1 |
| O | 38.8 |
| Se | 7.7 |

Ratio of Bi: O: Se = 4.3: 5: 1

This suggests near 1.075:1 ratio of Bi2O3: Bi2O2Se in this sample.