Nitrite anodic oxidation at Ni(II)/Ni(III)-decorated mesoporous SnO2 and its analytical

applications

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## Supplemental files

Fig. S1 High-resolution XPS spectra recorded in the Ni  $2p_{3/2}$  (a) and Sn 3d (b) regions for Ni/SnO<sub>2</sub> and in the C 1s region for Black Pearls (c). Nickel spectrum was fitted by overlapping Gaussian-Lorentzian curves assigned to NiO (1), Ni<sub>2</sub>O<sub>3</sub> (2) and Ni(OH)<sub>2</sub> (3). S and EL stand for the associated shake-up satellites and energy loss features, respectively.

The narrow scan XPS spectrum recorded in the Ni  $2p_{3/2}$  region (Fig. S1a) was successfully deconvoluted into three peaks located at binding energies of 854.5 eV, 855.8 eV

and 856.4 eV, ascribed to the presence on the surface of NiO, Ni<sub>2</sub>O<sub>3</sub> and Ni(OH)<sub>2</sub> species, respectively [1S,2S]. It was found that, in terms of relative surface concentration, NiO prevails (52.5%) whereas the presence of Ni<sub>2</sub>O<sub>3</sub> and Ni(OH)<sub>2</sub> is less marked (28.9% and 18.6%, respectively). In the Sn 3d region (Fig. S1b), the XPS spectra was identical to that of pristine SnO<sub>2</sub>, and the results obtained for the carbonaceous component (BP) (Fig. S1c) revealed only the presence of sp<sup>2</sup> carbon species [3S, 4S].



Fig. S2 Nitrogen adsorption-desorption isotherms for Ni/SnO<sub>2</sub> (a), Ni/SnO<sub>2</sub>-BP (b) and BP (c). Insets show the corresponding pore size distribution.

Nitrogen adsorption-desorption isotherms from Fig. S2 allowed the estimation of BET surface areas of *ca*. 36 m<sup>2</sup> g<sup>-1</sup> and *ca*. 115 m<sup>2</sup> g<sup>-1</sup> for Ni/SnO<sub>2</sub> and Ni/SnO<sub>2</sub>-BP, respectively whereas a value *ca*. 1488 m<sup>2</sup> g<sup>-1</sup> was found for the BP component. For both Ni-based materials the corresponding pore size distribution (insets in Figs. S2a and S2b) indicates the presence of mesopores with an average diameter of *ca*. 18 nm, although BP embedment results in a small additional contribution of larger pores, of *ca*. 45 nm diameter, most likely as a result of the higher average pore diameter of the BP material (see the inset in Fig. S2c)



Fig. S3 SEM micrographs for Ni/SnO<sub>2</sub> (a) and Ni/SnO<sub>2</sub>-BP (b). Insets show corresponding images at higher magnification.

## References

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