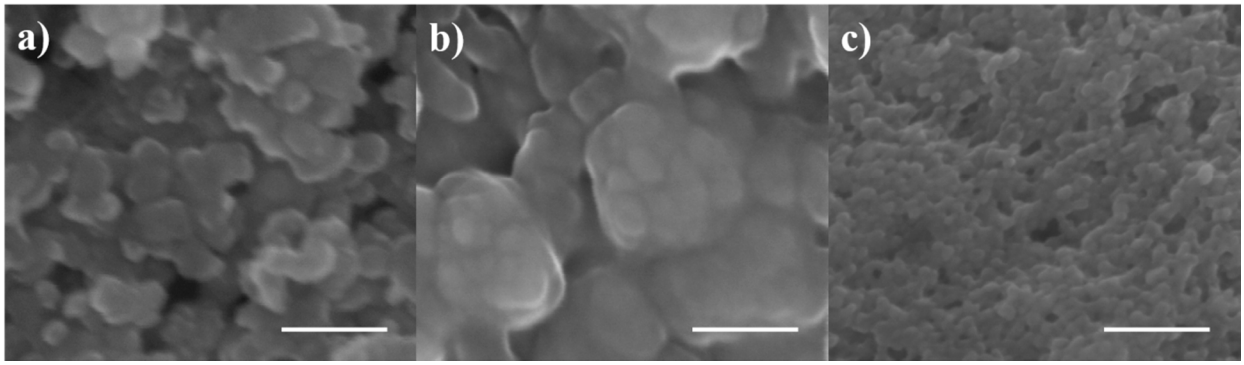

Figure S1. SEM images of (**a**) bare carbon screen-printed electrode (SPE), (**b**) Prussian Blue film on SPE, (**c**) PB-NiHCF nanoparticles on SPE. The scalebar is 100 nm.

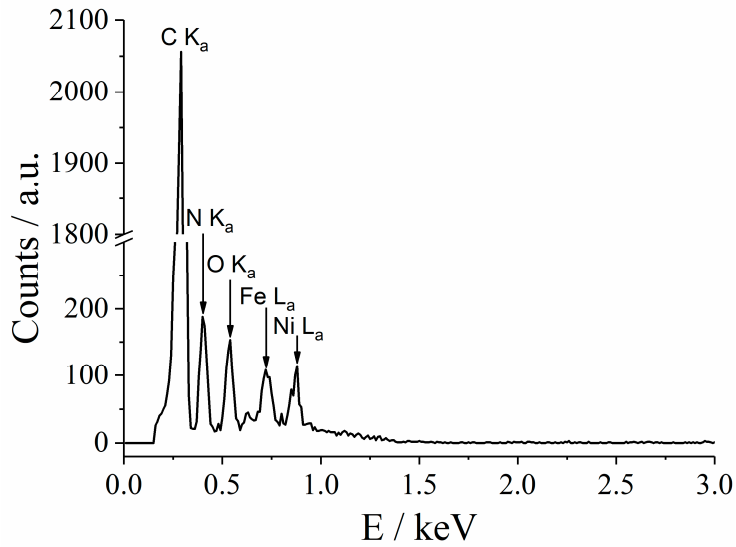

Figure S2. Energy dispersive X-ray (EDX) spectra of a PB-NiHCF nanoparticles modified carbon screen-printed electrode.

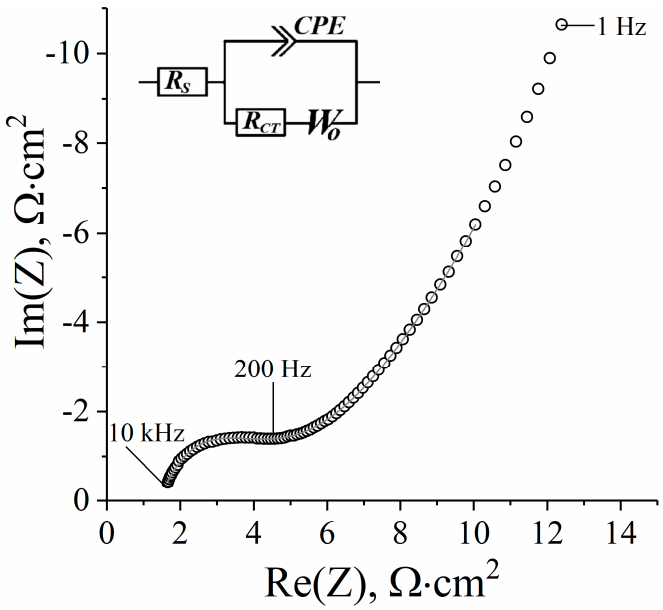

Figure S3. Impedance spectra in Niquist's plots of PB-NiHCF nanoparticles modified electrodes; 0.1 M KCl in 0.1 M HCl, ΔE = 5 mV, E_{DC} = 120 mV vs. Ag|AgCl (Prussian Blue|Prussian White redox potential). Solid lines represent fitting to the equivalent circuit shown in the inset.

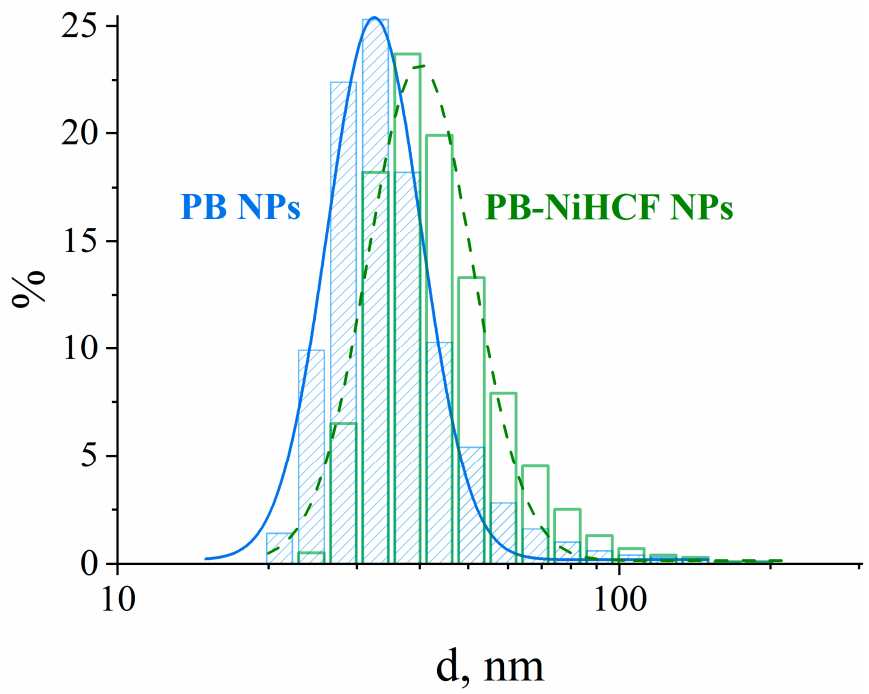

Figure S4. Dynamic light scattering fit to the log-normal distribution for Prussian Blue (—) and PB-NiHCF (- -) nanoparticles.

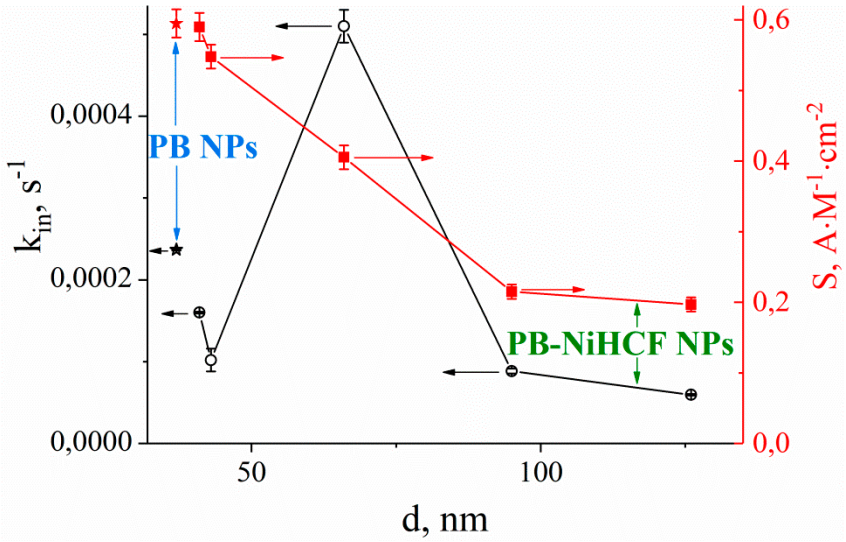

Figure S5. Sensitivities (■,★) and inactivation constants (○,☆) for sensors based on PB-NiHCF nanoparticles and Prussian Blue nanoparticles, respectively (0.5 mM H_2O_2, 0.1 M KCl, 0.05 M KH_2PO_4/ K_2HPO_4, pH 6.0, 0.0 V vs. Ag|AgCl).

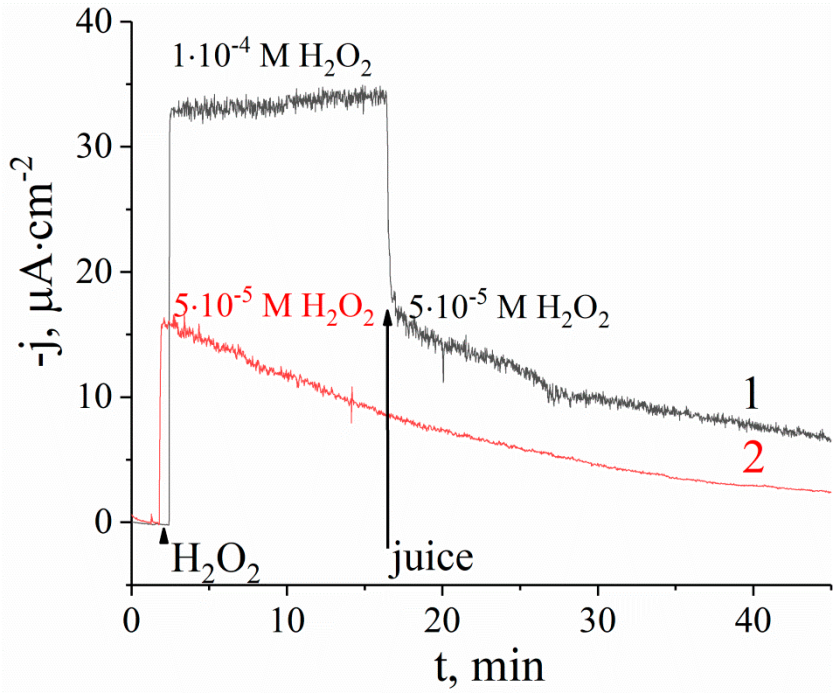

Figure S6. Amperometric signal of hydrogen peroxide sensor after H_2O_2 injection in phosphate buffer solution with consequent addition of fresh grapefruit juice (1) and after H_2O_2 injection in fresh grapefruit juice (2) (0.1 M KCl, 0.05 M KH_2PO_4/ K_2HPO_4, pH 6.0, 0.0 V vs. Ag|AgCl).

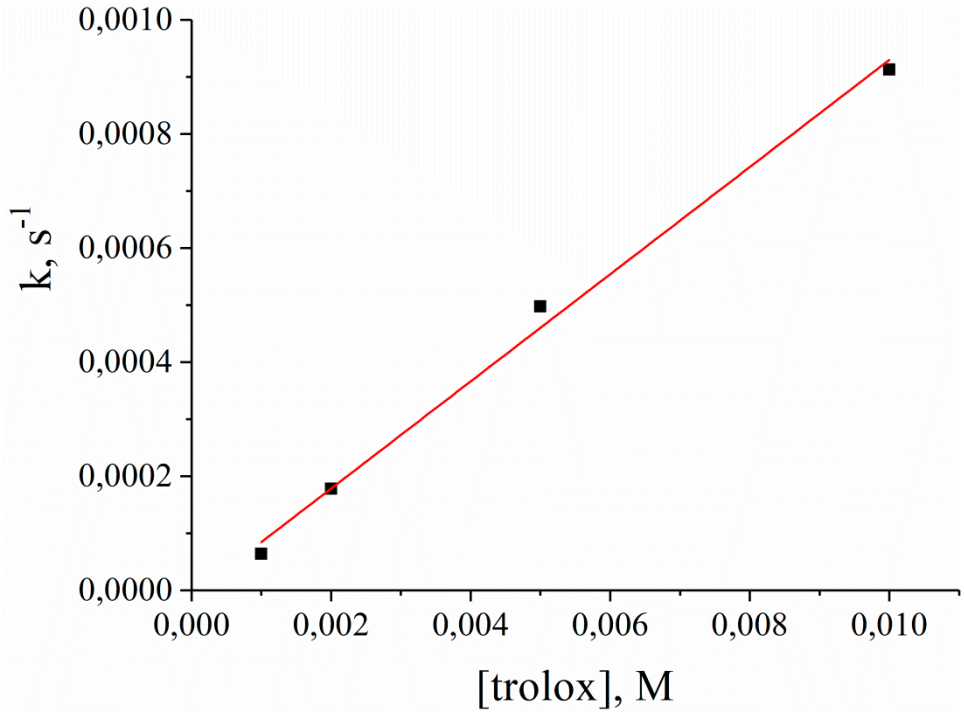

Figure S7. Dependence of antioxidant activity on trolox concentration (0.1 M KCl, 0.05 M KH_2PO_4/ K_2HPO_4, pH 6.0, 0.0 V vs. Ag|AgCl).