Supporting information for

Enhanced light-harvesting efficiency in Au metal-$NiFe_2O_4$ semiconductor hetero-nanostructures with implications for photoelectrochemical sensor towards the sensitive detection of Paracetamol in human urine

Tuan Anh Nguyen[1,*], Ngo Thi Loan[1,2], Thi Ngoc Mai Pham[3], Pham Duc Thang[4], Vu Ngoc Phan[1], Ngo Xuan Dinh[1], Tien Van Manh[5], Ong Van Hoang[5,1], Vu Dinh Lam[6], Anh-Tuan Le[1,2,**]

(1) Phenikaa University Nano Institute (PHENA), PHENIKAA University, Hanoi 12116, Vietnam

(2) Faculty of Materials Science and Engineering, PHENIKAA University, Hanoi 12116, Vietnam

(3) Faculty of Chemistry, VNU University of Science, Vietnam National University, Hanoi, 19 Le Thanh Tong, Hoan Kiem, Hanoi, 11000, Viet Nam

(4) Faculty of Physics, Center of Materials Science, VNU University of Science, Vietnam National University, Hanoi, 334 Nguyen Trai, Thanh Xuan, Hanoi, 11416, Viet Nam

(5) University of Transport Technology, Trieu Khuc, Thanh Xuan District, Hanoi, Viet Nam

(6) Graduate University of Science and Technology (GUST) & Institute for Materials Science (IMS), Vietnam Academy of Science and Technology, 18 Hoang Quoc Viet, Hanoi 10000, Vietnam

Corresponding authors:

*anh.nguyentuan1@phenikaa-uni.edu.vn (T-A. Nguyen)

**tuan.leanh@phenikaa-uni.edu.vn (A-T. Le)

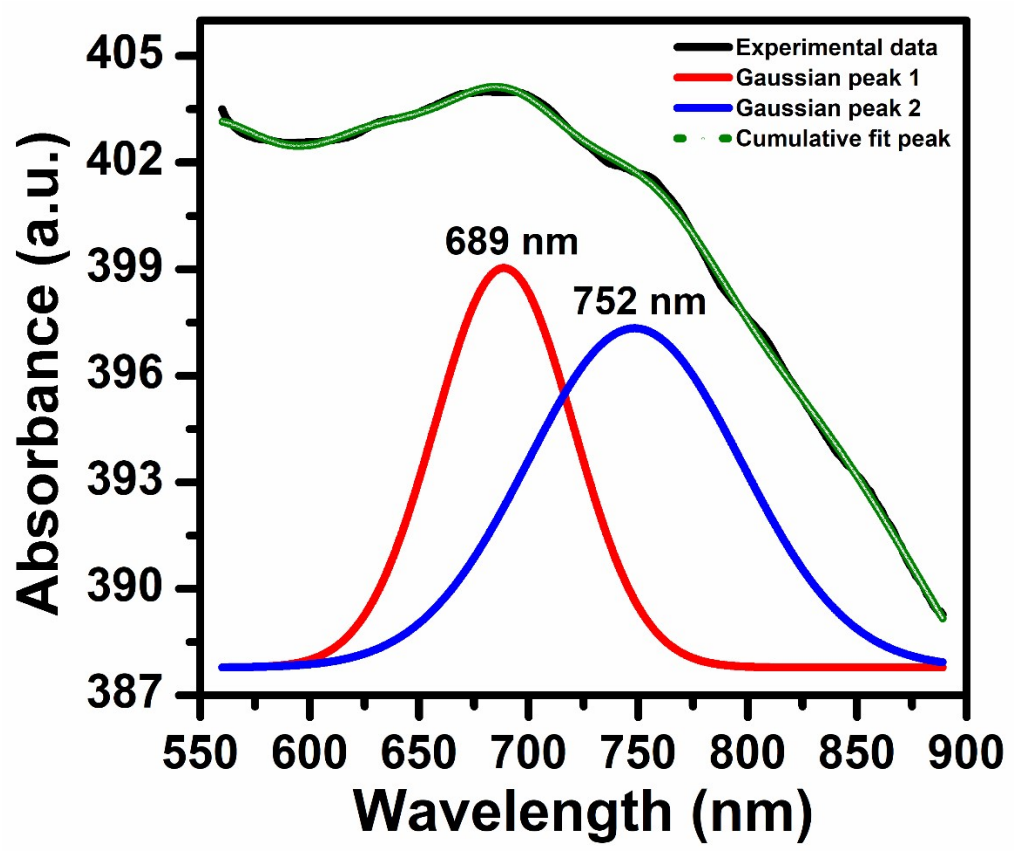

Fig. S1. UV-Vis adsorption spectra and their Gaussian fit bands of NFO nanoflakes

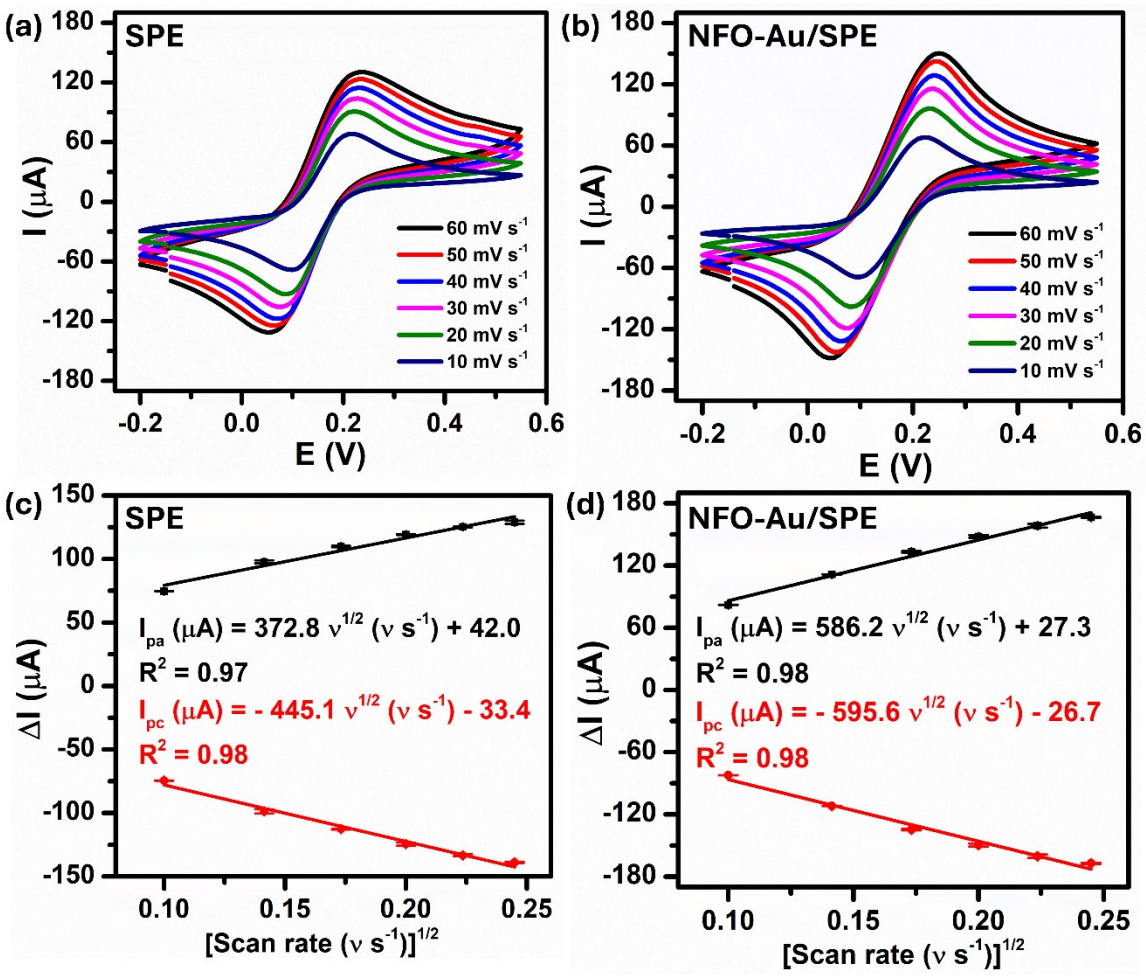

Fig. S2. (a and b) CV curves of the bare SPE and NFO-Au/SPE in 0.1 M KCl solutuion containing 5 mM $[Fe(CN)_6]^{3-/4-}$ at different scan rates and (c and d) linear plots for square root of scan rate versus oxidation/reduction peak currents with error bars, respectively.

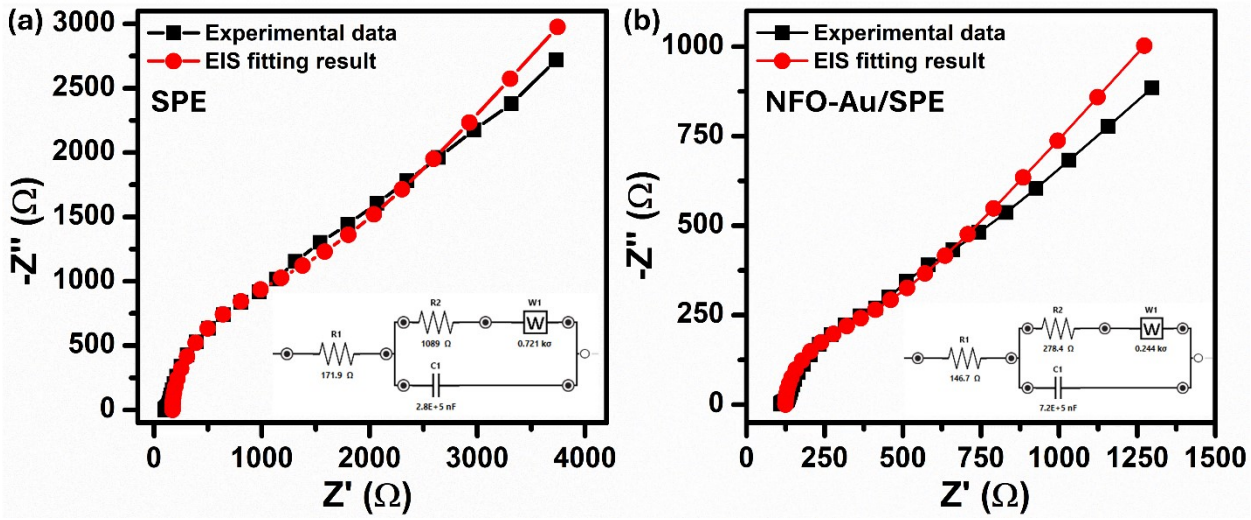

Fig. S3. Experimental data and fitted Nyquist plots of the bare SPE (a) and NFO-Au_4/SPE (b) in the frequency range from 0.01 kHz to 1000 kHz. Inset shows the Randles equivalent circuit used for fitting the data.

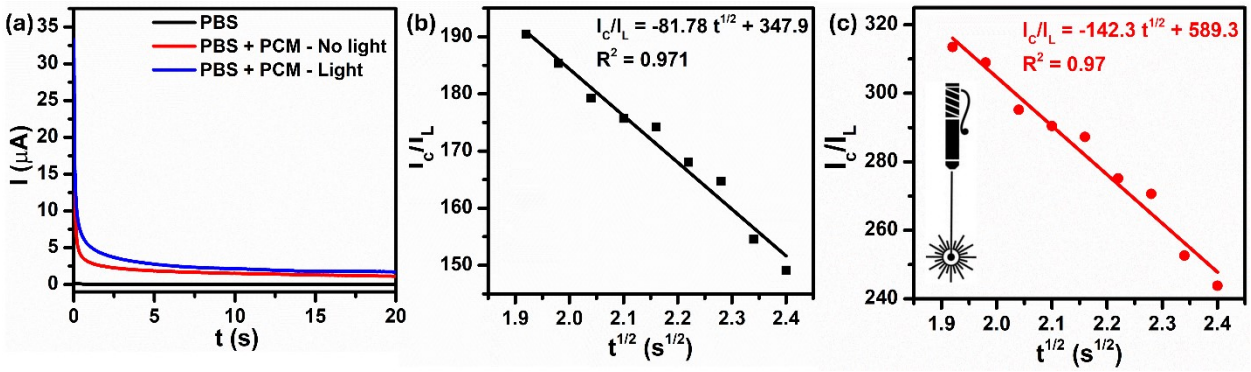

Fig. S4. (a) Chronoamperometric response of NFO-Au/SPE in PBS and in PBS containing 200 μM PCM in the absence and presence of visible light and (b and c) the plot of I_C/I_L *vs.* $t^{1/2}$ acquired from the chronoamperograms.

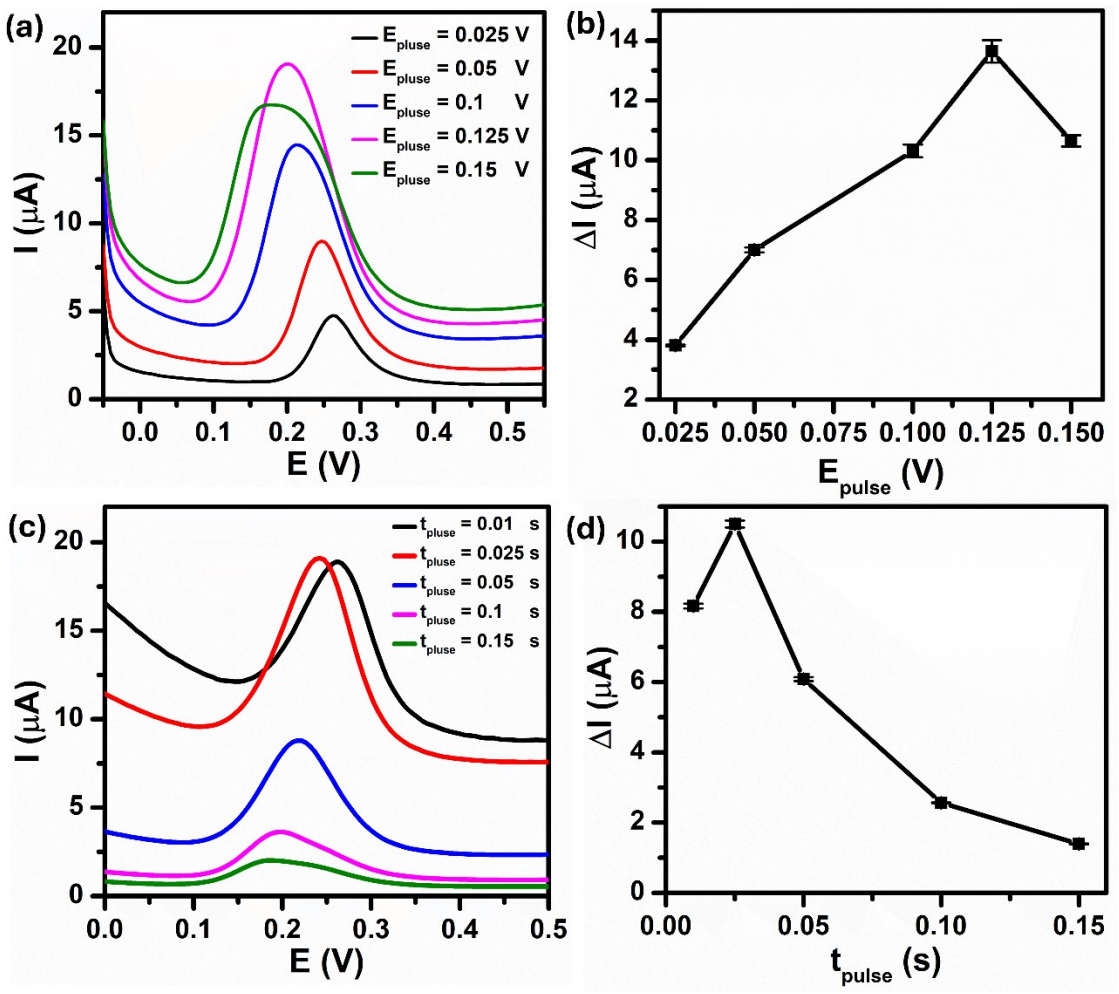

Fig. S5. DPV curves and the corresponding calibration plots of oxidation peak currents of 100 μM PCM record at NFO-Au/SPE with various pulse potentials (a and b) and pulse times (c and d), respectively.

Table S1 *Comparative study of the performance of various modified electrodes for PCM electrochemical detection* [a]

Modified Electrodes	**Techniques**	**Sensitivity ($\mu A\ \mu M^{-1}$)**	**Analytical ranges (µM)**	**Limit of detection (µM)**	**Ref.**
C60/GCE	DPV	0.013	50 – 1500	5	[1]
PAY/nano TiO_2/GCE	DPV	0.05	12 - 120	2.0	[2]
AuNP-PGA/SWCNTs	DPV	0.235	8.3 – 145.6	1.18	[3]
Fe_2O_3/CPE	DPV	0.017	2 – 150	1.16	[4]
ZrO_2/CPE	CV	0.0164	10 – 60	0.68	[5]
Ni-Al-HCF	Amperometry	0.0118	3 – 1500	0.8	[6]
$ZnFe_2O_4$/SPE	DPV	0.056	0.5 – 400	0.29	[7]
NFO-Au/SPE	DPV	0.196	0.5 – 200	0.37	This work

[a] GCE: glassy carbon electrode; PAY : poly(acid yellow 9); PGA: poly-glutamic acid;; SWCNTs: single-walled carbon nanotubes; CPE: carbon paste electrode; Ni-Al-HCF: hexacyanoferate(III) intercalated Ni Al layered double hydroxide

Table S2. *Recovery tests of PCM in human urine samples using NFO-Au-based electrochemical sensor under visible light irradiation.*

Sample No.[a]	Spiked values (µM)	Found values (µM)	Recovery (%)	RSD (%)
1	50	49.3	98.6	2.0
2	25	23.3	93.2	0.6
3	5	4.6	92.0	0.8

[a] Each sample was assayed in triplicate (n = 3).

References

1 R. N. Goyal and S. P. Singh, *Electrochim Acta*, 2006, **51**, 3008–3012.

2 S. A. Kumar, C.-F. Tang and S.-M. Chen, *Talanta*, 2008, **76**, 997–1005.

3 M.-P. N. Bui, C. A. Li, K. N. Han, X.-H. Pham and G. H. Seong, *Sens Actuators B Chem*, 2012, **174**, 318–324.

4 M. M. Vinay and Y. A. Nayaka, *Journal of Science: Advanced Materials and Devices*, 2019, **4**, 442–450.

5 S. B. Matt, S. Raghavendra, M. Shivanna, M. Sidlinganahalli and D. M. Siddalingappa, *J Inorg Organomet Polym Mater*, 2021, **31**, 511–519.

6 K. Asadpour-Zeynali and R. Amini, *Electroanalysis*, 2017, **29**, 635–642.

7 N. T. Anh, L. M. Tung, L. K. Vinh, N. Van Quy, O. Van Hoang, N. X. Dinh and A.-T. Le, *Nanoscale Adv.*, 2024, **6**, 256–267.