

## Electronic Supplementary Material

### **Preparation of thiol-grafted poly(3,4-ethylenedioxythiophene)/yolk shell carbon sphere/Au composites for the simultaneous detection of caffeic acid and levofloxacin**

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### 2.2.1 Synthesis of YRFC

The YRFC was prepared according to a previous literature procedure[25]. First, resorcinol (0.2 g) was injected into a flask containing 8.0 mL ethanol and 20 mL deionized water, and ultrasonically dispersed for 5 min. Then, 0.54 mL of formaldehyde was added to the above solution. After stirring for 10 minutes, the solution was transferred to a cryostat, and 0.3 mL ammonia water was slowly added dropwise. The solution was stirred at 0 °C for 24 h, and the obtained milky white solution was washed 3 times alternately with water and ethanol. Then, it was freeze-dried for 12 h. A resorcinol and formaldehyde resin with a yolk-shell structure was obtained, which is represented by YRF. The dried powder was placed into a porcelain boat, heated to 700 °C for carbonization at a heating rate of 1.5/min, and then held at 700 °C for 1 h. The prepared sample was denoted as YRFC.

### 2.2.3 Synthesis of Au NPs

The preparation process of Au NPs is as follows: Take 0.314 mL of 0.048 mol / L  $\text{HAuCl}_4 \cdot 3\text{H}_2\text{O}$  solution and place it in a reaction flask containing 50 mL of distilled water. After boiling, quickly add 0.75 mL of 1% (w/v)  $\text{C}_6\text{H}_5\text{Na}_3\text{O}_7$  solution to the boiling in the solution, while reflux and vigorously stir. The color changed from light yellow to transparent red wine within 3 minutes. The reflux continued for 10 minutes before continuously stirring the product and cooling it to room temperature. Colloidal gold nanoparticles (Au NPs) were obtained.

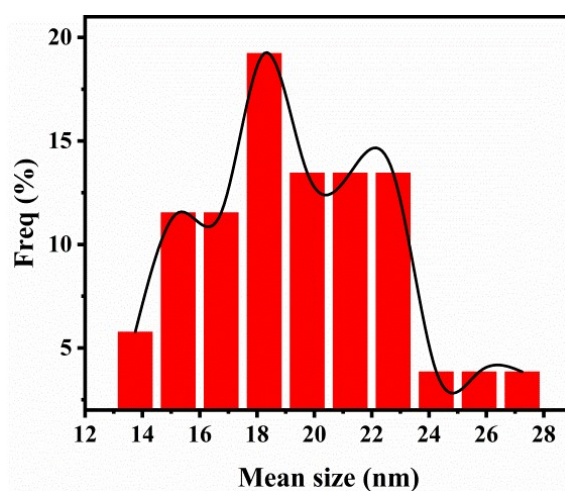


Fig. S1. The mean size distribution of Au NPs in the PEDOTMeSH/YRFC/Au

Table S1. The  $K_{\text{et}}$  values of unmodified and modified electrodes

Electrodes	A ( $\text{cm}^2$ )	$R_{\text{ct}}$ ( $\Omega$ )	$k_{\text{et}}$ ( $\text{cm s}^{-1}$ )
Bare GCE	0.1256	274	$1.6 \times 10^{-3}$
YRFC/Au/GCE	0.1256	186	$2.24 \times 10^{-3}$
PEDOT/YRFC/Au/GCE	0.1256	44	$9.6 \times 10^{-3}$

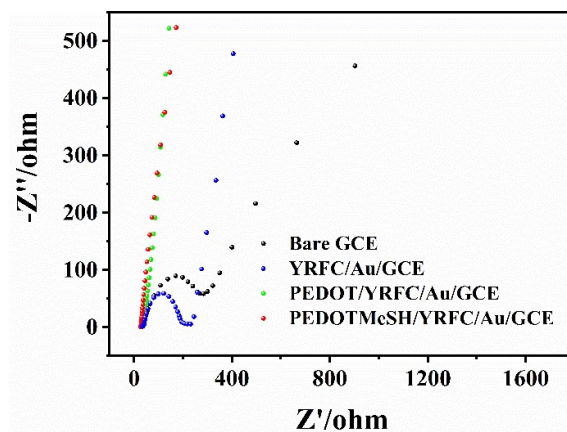


Fig S2. EIS Nyquist plots of unmodified and modified electrodes

Table S2. The analytical parameters for individual and simultaneous detection of PEDOTMeSH/YRFC/Au/GCE

Analyte	Modified electrode	Linear range/ $\mu\text{molL}^{-1}$	LOD/ $\mu\text{molL}^{-1}$	$y=ax + b$	$R^2$
Individual detection	PEDOTMeSH/YRFC/Au/GCE	0.02-310	0.011	$y = 0.1618x + 8.0819$	0.9982
		0.06-300	0.019	$y = 0.0904x + 3.8431$	0.9967
Simultaneous detection	PEDOTMeSH/YRFC/Au/GCE	0.02-320	0.014	$y = 0.1254x + 1.9927$	0.9961
		0.02-320	0.018	$y = 0.0948x + 2.6177$	0.9962

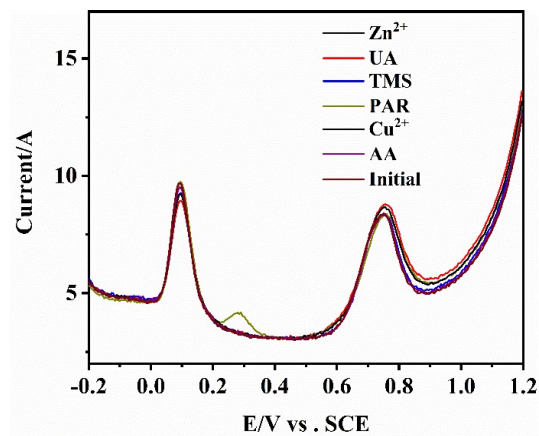


Fig. S3. The DPV curves of different interferences of PEDOTMeSH/YRFC/Au/GCE in 0.1M PBS

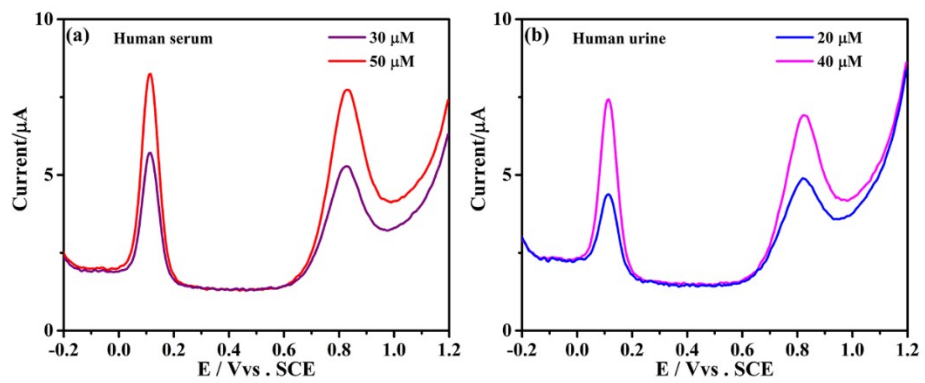


Fig. S4. Simultaneous detection DPV response of PEDOTMeSH/YRFC/Au/GCE in human serum and human urine