

Supplementary material to NiFe₂O₄/(S,N)-doped graphene oxide composite as efficient electrochemical sensor for determination of sibutramine in dietary supplements

Ngo Thi Thanh Xuan^{1,2} , Le Thi Thanh Nhi^{3,4} , Nguyen Quang Man⁵ ,
Vo Thi Khanh Ly⁵ , Le Thi Hong Phong⁶ , Nguyen Hai Phong¹ , Pham Khac Lieu¹ ,
Le Lam Son¹  and Dinh Quang Khieu¹ 

¹University of Sciences, Hue University, 530000, Vietnam

²Drug, Cosmetic and Food Quality Control Center of Hue City, 530000, Vietnam

³Center for Advanced Chemistry, Institute of Research and Development, Duy Tan University, 03 Quang Trung, Da Nang 550000, Vietnam

⁴Faculty of Natural Sciences, Duy Tan University, 03 Quang Trung, Da Nang 550000, Vietnam

⁵University of Medicine and Pharmacy, Hue University, 530000, Vietnam

⁶Institute of Materials Sciences, Vietnam Academy of Science and Technology, 18 Hoang Quoc Viet, Cau Giay, Hanoi City 100000, Vietnam

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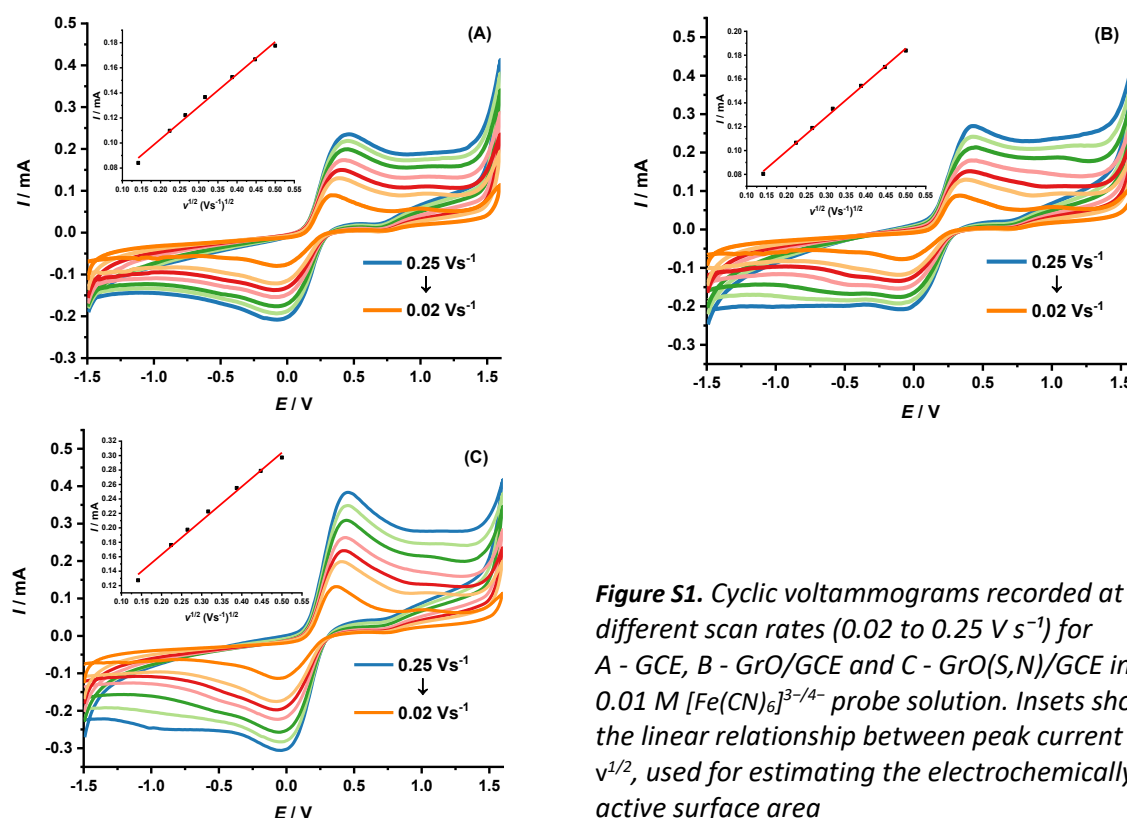


Figure S1. Cyclic voltammograms recorded at different scan rates (0.02 to 0.25 V s⁻¹) for A - GCE, B - GrO/GCE and C - GrO(S,N)/GCE in 0.01 M [Fe(CN)₆]^{3-/4-} probe solution. Insets show the linear relationship between peak current and v^{1/2}, used for estimating the electrochemically active surface area

The electrochemically active surface area increased from 0.0348 cm² (GCE) and 0.0387 cm² (GrO/GCE) to 0.0634 cm² for GrO(S,N)/GCE, indicating that S,N co-doping significantly increased the number of accessible electroactive sites.

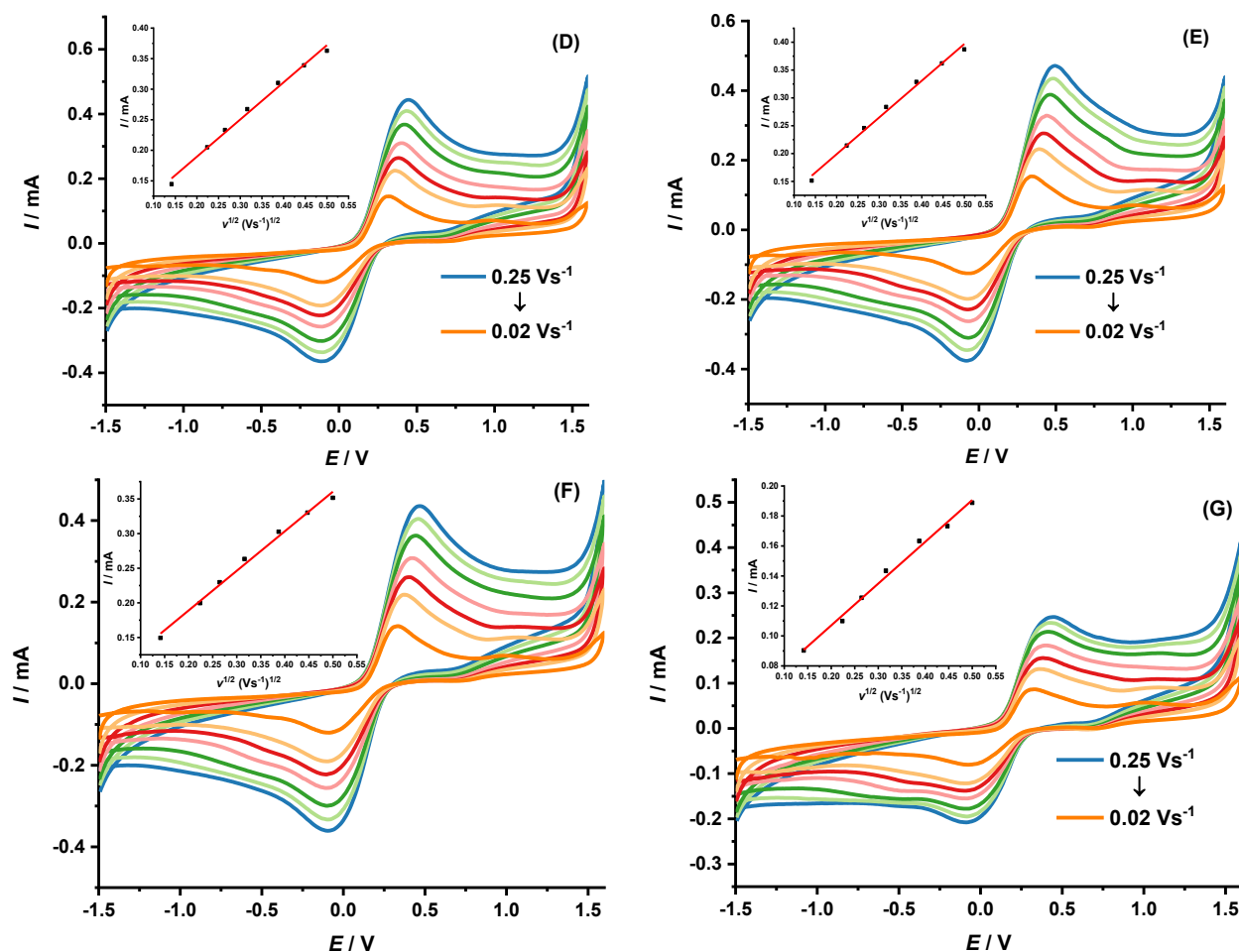


Figure S2. Cyclic voltammograms recorded at different scan rates (0.02 to 0.25 V s⁻¹) for A - (0.5/1)NiF/GrO(S,N), B - (1/1)NiF/GrO(S,N), C - (1.5/1)NiF/GrO(S,N) and D - NiF in 0.05 M [Fe(CN)₆]^{3-/4-} probe solution. Insets show the linear relationship between peak current and $v^{1/2}$, used for estimating the electrochemically active surface area

The calculated ECSA values were 0.0376, 0.0820, 0.0890, and 0.0771 cm² for NiF, (0.5/1)NiF/GrO(S,N), (1.0/1)NiF/GrO(S,N) and (1.5/1)NiF/GrO(S,N), respectively, with the highest value obtained for (1.0/1)NiF/GrO(S,N).

The peak currents of STB reached relatively high values at 0.4 V and remained stable compared to other potentials. Therefore, 0.4 V was selected as the optimal accumulation potential for subsequent measurements to ensure both sensitivity and signal stability (Figure S3).

The peak currents of STB increased with increasing accumulation time and reached a maximum at 5 s, after which a slight decrease was observed, likely due to surface saturation effects (Figure S4).

The peak currents increased with increasing pulse amplitude and reached maximum values at 0.1 V. A further increase resulted in signal instability; therefore, 0.1 V was selected as the optimal pulse amplitude for subsequent measurements (Figure S5).

Although higher peak currents were observed at 0.06 to 0.8 V, the signals became less stable. Therefore, 0.07 V was selected as the optimal pulse amplitude, providing a good compromise between sensitivity and signal stability (Figure S6).

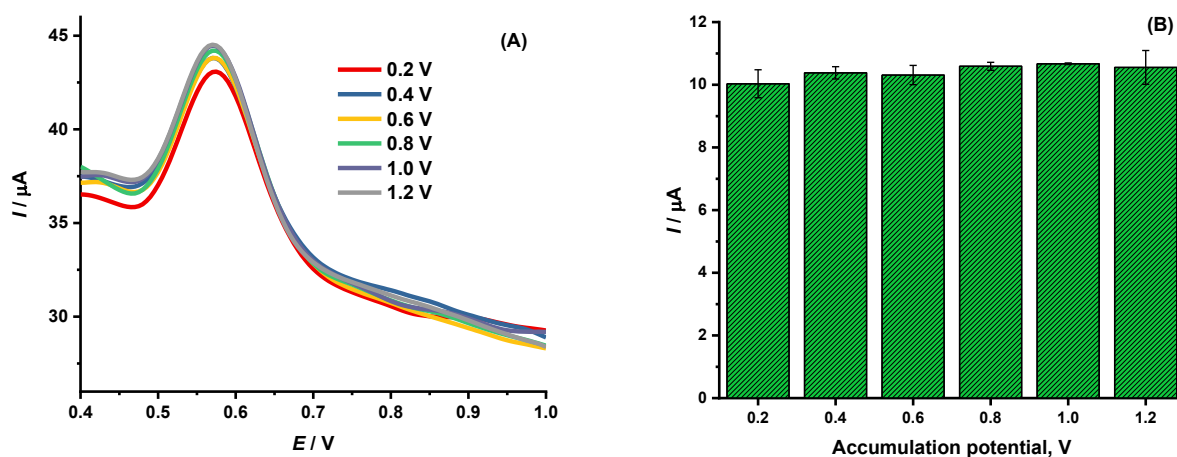


Figure S3. A - DPV curves of (1.0/1)NiF/GrO(S,N)/GCE recorded in 0.1 M BRS (pH 3) containing 10 μ M STB at different accumulation potentials (0 to 1.2 V). Experimental conditions: accumulation time 5 s; pulse amplitude 0.06 V; voltage step 0.005 V, B - corresponding peak currents of STB as a function of accumulation potential

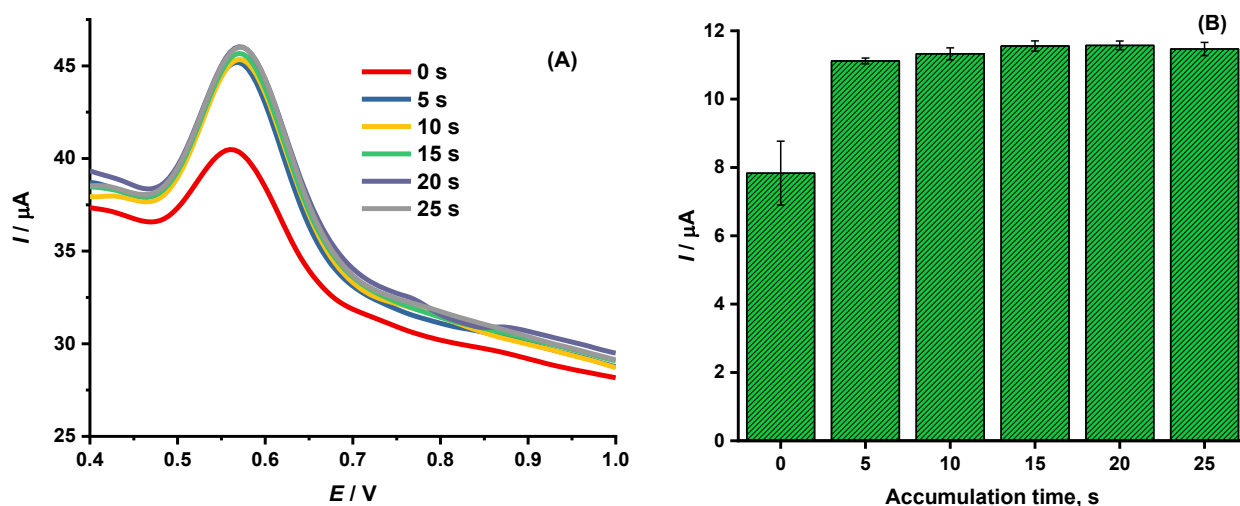


Figure S4. A - DPV curves (1.0/1)NiF/GrO(S,N)/GCE recorded in 0.1 M BRS (pH 3) containing 10 μ M STB at different accumulation times (0 to 25 s). Experimental conditions: accumulation potential 0.4 V; pulse amplitude 0.06 V; voltage step 0.005 V, B - corresponding peak currents of STB as a function of accumulation time

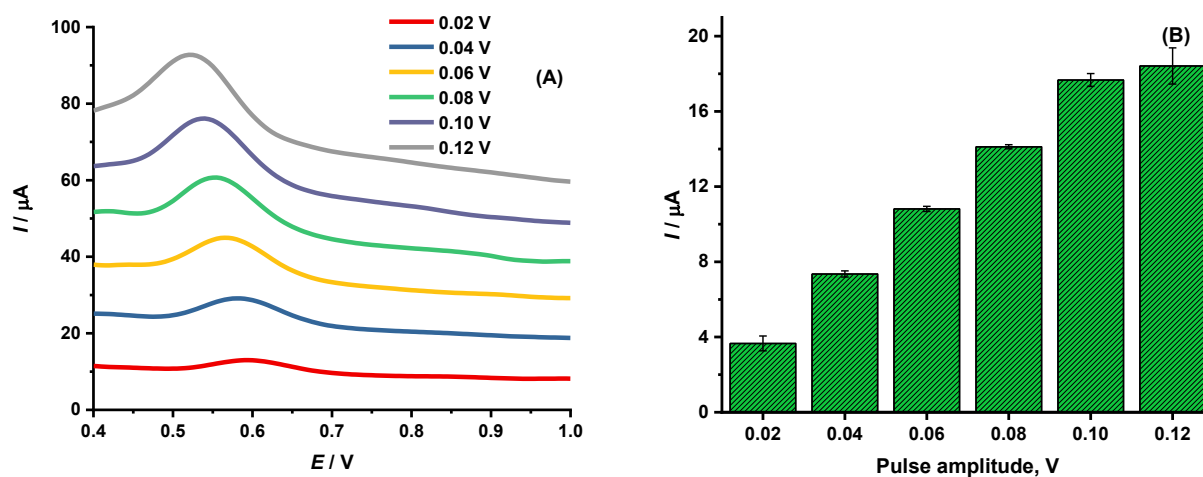


Figure S5. A - DPV curves of (1.0/1)NiF/GrO(S,N)/GCE recorded in 0.1 M BRS (pH 3) containing 10 μ M STB at different pulse amplitudes (0.02 to 0.12 V). Experimental conditions: accumulation potentials 0.4 V; accumulation time 5 s; voltage step 0.005 V, B - corresponding peak currents of STB as a function of pulse amplitude

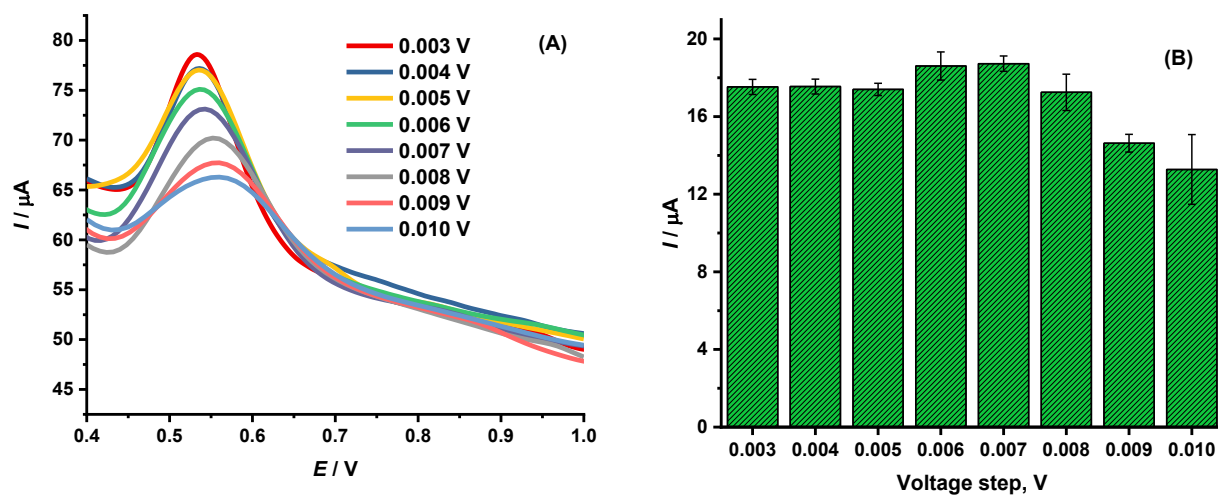


Figure S6. A - DPV curves of (1.0/1)NiF/GrO(S,N)/GCE recorded in 0.1 M BRS (pH 5.0) containing 10 μ M STB at different voltage steps (3 to 10 mV). Experimental conditions: accumulation time 5 s; accumulation potential 0.4 V; pulse amplitude 0.1 V, B - corresponding peak currents of STB as a function of voltage steps