A novel synthesis and investigation of electrochemical behaviors of HPNPC/GC sensor electrode: Using simultaneously determination of dopamine, ascorbic acid and uric acid

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Modification of carbon surfaces by electrochemical techniques is an important objective in electrochemistry and material science. In electrochemical techniques, carbon electrodes are widely used because of low cost, low equipment, low background current, wide potential window, speed, chemical inertness and minimum sample pretreatment required prior to analysis (Kuhnau, 1976; Nematollahi and Malakzadeh, 2003). One of the reasons for the considerable current interest in modification of carbon materials is because the carbon material is inert and has high conductivity and resistance to environmental and chemical attack. Electrochemical techniques are based on the direct reduction or oxidation of substrate onto an electrode surface. Reactions onto the electrode surfaces are very suitable for analytical applications due to their requirements of high potential. Moreover, these surfaces can be modified by a reductive substrate for analytical applications.

**Synthesis of 4-hydroxyphenethyl-4-(4-nitrophenyl)piperazine-1-carbodithioate (HPNPC)**

A mixture of compound NaOH (3.0 mmol), appropriate piperazine/piperidine derivative (1.0 mmol) (1-(4-nitrophenyl) piperazine) and CS₂ (5.0 mmol) in ethanol, was refluxed for 1 h at room temperature (Yurttaş et al., 2016). After that, the mixture was filtered to remove solvent. The residue was treated with 25 mL of ether. Solidified product was filtered, washed with water, and recrystallized from ether to give the compounds. The synthesized compound (1.0 mmol) was dissolved in acetone and added 4-hydroxy-phenylethyl bromide (1.0 mmol) and K₂CO₃ (1.2 mmol) was refluxed for 2 h at 40 °C. After cooling, the solvent was evaporated until dryness and recrystallized from ethanol to give the compounds (Yurttaş et al., 2014). Yield: 69%. m.p. 197.5°C. FTIR (ATR) cm⁻¹: 3394 (O–H), 1577–1425 (C=C), 1516–1303 (NO₂), 821 (1,4-disubstituted benzene). ¹H-NMR (500 MHz, DMSO-d₆): δ = 2.84 (2H, t, J=8.00 Hz, -CH₂-), 3.46 (2H, t, J=7.50, CH₂), 3.71 (4H, t, J=5.50 piperazine CH₂), 4.09 (2H, s, piperazine CH₂), 4.39 (2H, s, piperazine CH₂), 6.71(2H, d, J=8.50, 1,4-phenyl), 6.95 (2H, d, J=9.50, 1,4-phenyl), 7.09 (2H, d, J=8.00, 1,4-phenyl), 8.11 (2H, d, J=9.50, 1,4-phenyl), 9.25 (1H, s, OH). ¹³C-NMR (125 MHz, DMSO-d₆): δ = 34.11, 38.18, 51.19, 53.17, 112.32, 115.66, 126.26, 129.87, 130.61, 137.35, 154.21, 153.35, 195.97. HRMS (m/z): [M+H]+ calculated for C₁₉H₂₁N₃O₃S₂: 404.1097; found 404.1098.

**Scheme 1.** Synthetic pathway for the synthesis of the target compound.

**Scheme 2.** Modification mechanism of HPNPC at GC electrode surface (in CH₃CN containing 100 mM NBu₄BF₄).

Electrochemical and spectroelectrochemical properties of newly synthesized 4-hydroxyphenethyl-4-(4-nitrophenyl)piperazine-1-carbodithioate (HPNPC) were studied using glassy carbon (GC) electrode. In the current study, surface characterization was performed by cyclic voltammetry (CV) besides electrochemical impedance spectroscopy (EIS) was used for the electrochemical and spectroelectrochemical characterization. Surface images were obtained using scanning electron microscopy (SEM). After completion of modification and characterization...
process, HPNPC/GC surface was examined for the usability for the quantitative determination of dopamine, ascorbic acid and uric acid by using CV, square wave voltammetry (SWV) and differential pulse voltammetry (DPV). Dopamine, ascorbic acid and uric acid solutions that were prepared in phosphate buffer solution (pH 6.80) were employed in this research. The presented study proves that all of compounds can be quantitatively determined not only simultaneously but also independently.

References