



Green synthesized nickel nanoparticles modified electrode in ionic liquid medium and its application towards determination of biomolecules

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ABSTRACT

An air and moisture stable ionic liquid 1-ethyl-3-methylimidazolium ethyl sulfate (EMIMES) was used as an electrolyte for electropolymerization of L-cysteine followed by electrodeposition of nickel nanoparticles (NiNP) on paraffin wax impregnated graphite electrode (PIGE). The electrodeposited NiNP modified electrode showed good redox activity and stability in 0.1 M KOH solution. The modified electrode has been characterized using Fourier transform infrared spectroscopy (FTIR), Raman spectroscopy, X-ray photoelectron spectroscopy (XPS), field emission scanning electron microscopy (FESEM), energy dispersive X-ray spectroscopy (EDS), electrochemical impedance spectroscopy (EIS) and cyclic voltammetry (CV). The modified electrode was examined for electrocatalytic oxidation of some compounds of biological and clinical importance such as vitamin B₆, L-tyrosine, L-tryptophan, vanillin, glucose and hydrogen peroxide by cyclic voltammetry to demonstrate the electrocatalytic activity of the electrodeposited NiNPs.

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1. Introduction

Room-temperature ionic liquids (RTILs) are materials that are composed entirely of ions and are liquids below 100 °C [1]. The unique properties of RTILs make them particularly promising for electrochemical applications. RTILs are ionically conductive and hence electrochemistry can be performed using RTILs as supporting electrolytes. Many RTILs offer wide potential windows (the potential range over which the RTIL is electrochemically inert), thermal and chemical stability, very low vapor pressures, and the ability to dissolve a wide range of chemical species. Due to the unique combination of these properties, electrochemists have proposed the use of RTILs in a wide range of devices including photoelectrochemical cells [2], supercapacitors [3], fuel cells [4], batteries [5] and electrochemical sensors [6,7]. Modified electrodes offer higher selectivity, sensitivity, greater time efficiency and higher stability than the conventional electrodes. Therefore, several modifiers such as polymers [8–11], DNA doped polymers [12], dye doped sol-gels [13], metal oxides [14], carbon nanotubes [15,16], inorganic redox mediators [17] and metal nanoparticles (MNPs) [18–20] have been used to fabricate modified electrodes. Among them, MNPs modified electrodes have drawn particular attention due to their high surface area, effective mass transport, catalysis and control over local microenvironment compared to electrodes modified with macroparticles [21,22]. However, the MNPs at the electrode surface can be fragile in the absence of stabilizing conductive material. To address

this problem, the electrode surface can be modified with some conductive stabilizing materials such as carbon nanotubes and polymers [23,24] and then the MNPs can be attached to the modified electrode surface. Moreover, the porous nature of conducting polymer allows dispersing of the metal nanoparticles into the polymer matrix and generates additional electrocatalytic sites [25–27]. Polycysteine (PC) is one of the important polyaminoacid, its structure is relevant to biopolymers and it supports the metal nanoparticles. Platinum nanoparticles–polypyrrole modified electrodes have been employed to determine nitrite and to study proton and oxygen reduction [28,29]. To the best of our knowledge, there is no reported relevant literature available for the determination of biologically and clinically important compounds at polycysteine functionalized nickel nanoparticles (PCFNiNP) modified electrodes.

In the present study, a modified electrode was prepared by electrochemical deposition of NiNP in over-oxidized polycysteine film modified graphite electrode in ionic liquid medium and the performance of the electrode was studied by cyclic voltammetry and surface examination was made using FESEM. The PCFNiNP modified electrode was used as the sensing matrix for the determination of vitamin B₆, L-tyrosine, L-tryptophan, vanillin, glucose and hydrogen peroxide at micromolar levels.

2. Experimental

2.1. Reagents

Vitamin B₆, L-tryptophan, L-tyrosine, D-glucose, vanillin and hydrogen peroxide were purchased from HIMEDIA and dissolved

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before use. All other chemicals used in this investigation were of analytical grade. EMIMES (purity 98.5%) was obtained from Alfa Aesar, spectroscopic grade graphite rod (3 mm diameter) was used as received from Aldrich. Double distilled water employed in all experiments was obtained from a Millipore-Milli-Q system.

2.2. Apparatus and measurements

FTIR spectra were recorded using a Perkin–Elmer RX 1 spectrometer. Raman spectra were recorded with Raman 11i system (Nanophoton Corp., Japan). For the study of morphology and chemical composition of the PCFNiNP modified electrode, FE-

SEM and EDS data were obtained on a SU6600 field emission scanning electron microscopy (FE-SEM) (HITACHI, Japan), equipped with an energy-dispersive X-ray analyzer at an accelerating voltage of 30 kV. XPS measurement for surface analysis was performed on a monochromatic 300 W Al K α X-ray radiation as the X-ray source for excitation (Model XM 1000, Omicron Nanotechnology, Germany).

Electrochemical measurements were performed with a CHI 660B electrochemical workstation (CH Instruments, USA) controlled by a personnel computer. Three electrode system was employed for this study. A platinum wire and a saturated calomel electrode (SCE) were used as auxiliary and reference electrodes, respectively. All potentials were referred to the latter. The modified electrode employed as the working electrode was prepared according to the procedure described below. A magnetic Teflon stirrer provided the convective transport during the amperometric measurements. All electrochemical experiments were performed in 0.1 M KOH solutions.

2.3. Construction of the PCFNiNP modified electrode

PIGE with a circular surface diameter of 3 mm prepared as reported [30] was used for electrode modification. One end of the electrode was carefully polished on a smooth surface and with 0.5 μ m alumina, washed with distilled water and dried in air. Then, the polished surface was used for electrode modification. The electropolymerization of 0.01 M L-cysteine was carried out in 0.1 M EMIMES by cycling the potential between -0.6 to 2.0 V at 50 mV/s scan rate. A thin polycysteine (PC) formed with 15 deposition cycles was used in all experiments. After removing the electrode from depositing solution, it was washed with ultrapure

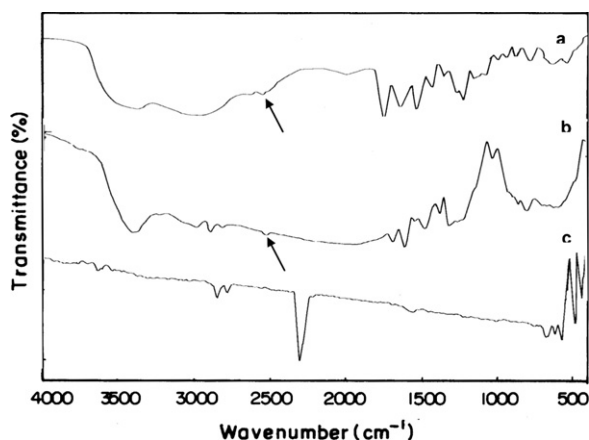


Fig. 1. FTIR spectrum of pure (a) L-cysteine, (b) PC modified electrode and (c) PCFNiNP modified electrode.

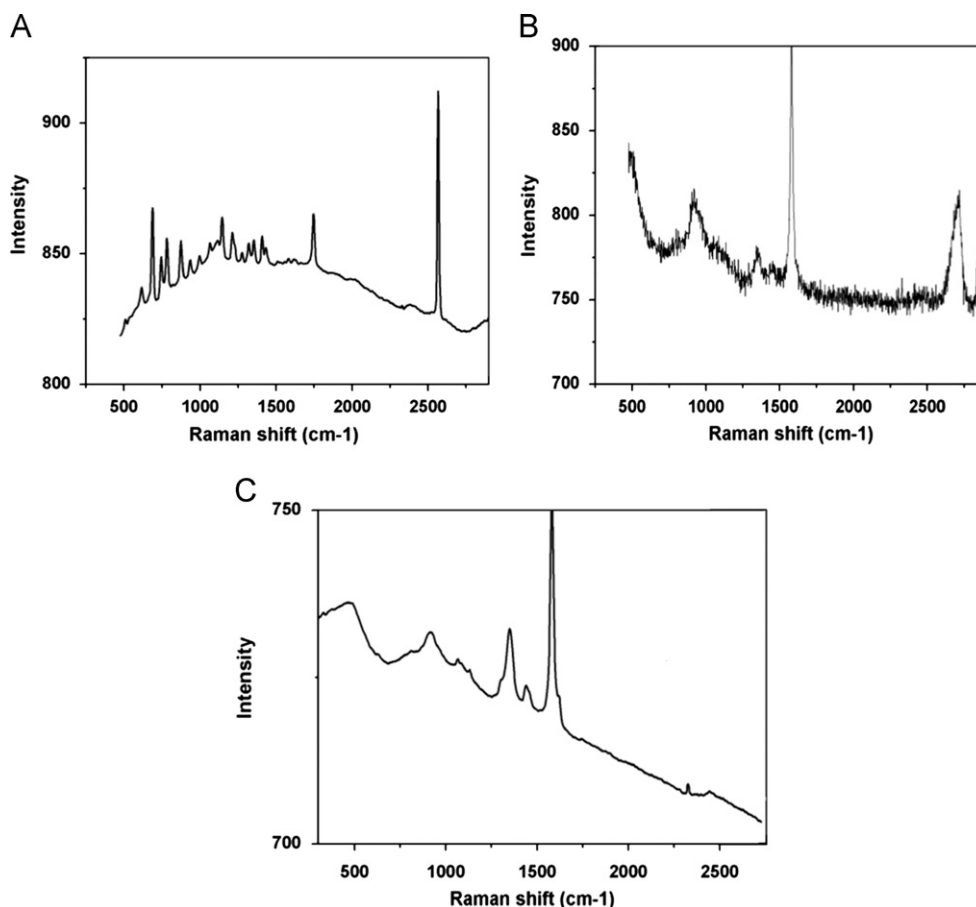


Fig. 2. Raman spectra of (A) pure L-cysteine, (B) PC modified electrode and (C) PCFNiNP modified electrode.

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