ABSTRACT

Presented Diploma Thesis deals with the study of electrochemical behavior of 5-nitroimidazole (5-NI), with the search for optimal conditions for its determination using techniques of DC voltammetry (DCV) and differential pulse voltammetry (DPV) at a mercury meniscus modified silver solid amalgam electrode (m-AgSAE) and at a boron-doped diamond film electrode (BDDFE), and with the comparison of reached limits of quantification (L_Qs).

The optimal conditions found for measuring calibration dependences of 5-NI at the m-AgSAE in deionized water were as follows: Britton-Robinson buffer of pH 7.0 and 10.0 for DCV and DPV, respectively. At both thechniques, it is advisable to use an electrochemical regeneration of the electrode surface by the application of a couple of regeneration potentials $E_{reg,1}$ = 0 mV and $E_{reg,2}$ = −800 mV. The calibration dependences were measured in the concentration range from $1{\cdot}10^{-6}$ to $1{\cdot}10^{-4}$ mol·l^{-1}, with the L_Qs of $1.0{\cdot}10^{-6}$ mol·l^{-1} for DCV at the m-AgSAE and $1.5{\cdot}10^{-6}$ mol·l^{-1} for DPV at the m-AgSAE.

The concentration dependences of 5-NI at the BDDFE were measured using the DCV technique in the BR buffer of pH 3.0. For the DPV technique, the optimal medium seemed to be the BR buffer of pH 2.0, however, because of the presence of an interfering peak in the supporting electrolyte, the concentration dependences were measured alternatively in the 0.1 mol l^{-1} acetate buffer of pH 4.6. For both techniques, optimal regeneration potentials were found as $E_{reg,1}$ = 0 mV and $E_{reg,2}$ = 1700 mV. At the measurement of the concentration ranges of 5-NI from $1{\cdot}10^{-6}$ to $1{\cdot}10^{-4}$ mol·l^{-1}, the L_Qs reached were $6.0{\cdot}10^{-7}$ and $1{\cdot}0{\cdot}10^{-6}$ mol·l^{-1} for DCV and DPV, respectively.

The applicability of the newly developed methods for the determination of 5-NI was verified on model samples of drinking and river water. The attained L_Qs of 5-NI at the m-AgSAE were in the concentration order of 10^{-6} mol·l^{-1} for both DCV and DPV. For DCV and DPV at the BDDFE, the L_Qs reached at given conditions were comparable.

Moreover, the mechanism of electrochemical reduction of 5-NI was investigated at both aforementioned working electrodes using cyclic voltammetry. It was found that the reduction of 5-NI is fully controlled by the diffusion only at the BDDFE, just in strongly alkaline media. In all other cases, the rate of the electrochemical process is partially influenced by the charge-transfer reaction kinetics.