Abstract

The aim of this Diploma Thesis was to develop sensitive, inexpensive, and less timeconsuming voltammetric methods for the determination of diacetyle and pyruvate, when both analytes are derivatized by *o*-fenylendiamine (OPDA) and the resulting reaction products are determined. For this purpose, techniques DC voltammetry (DCV) and differential pulse voltammetry (DPV) at a mercury meniscus modified silver solid amalgam electrode (m-AgSAE) were used.

The optimization consisted of finding a suitable medium and regeneration of the electrode surface for the determination 2,3-dimethylquinoxaline (2,3-DMQ) and 2-hydroxy-methylquinoxaline (2-OH-3-MQ), products of derivatization reactions. The optimal medium for the 2,3-DMQ determination was a solution of the Britton-Robinson buffer (BR-buffer) of pH 6.0 for DCV (optimal regeneration potentials $E_{in} = -400$ mV and $E_{fin} = -900$ mV, with limit of quantification $L_Q \approx 1.2 \cdot 10^{-6}$ mol·L⁻¹) and pH 7.0 for DPV ($E_{in} = -200$ mV and $E_{fin} = -900$ mV, $L_Q \approx 1.1 \cdot 10^{-7}$ mol·L⁻¹). For 2-OH-3-MQ, the optimal medium was BR-buffer of pH 4.0 and regeneration potentials $E_{in} = -500$ mV and $E_{fin} = -900$ mV for both techniques ($L_Q \approx 4.8 \cdot 10^{-7}$ mol·L⁻¹ for DCV and 3,4 $\cdot 10^{-7}$ mol·L⁻¹ for DPV).

Optimization of derivatization reactions for the determination of diacetyle and pyruvate consisted of finding the optimal concentration of OPDA, which was $9 \cdot 10^{-4}$ mol·L⁻¹, the pH of the reaction (pH = 1.2, adjusted by HCl), time (30 min) and temperature (35 °C). Post-derivatization measurements were made by DPV technique at the previously found optimal conditions for the determination.

For the determination of diacetyle and pyruvate in their mixture, new optimal conditions were found: BR-buffer pH 5.0, $E_{in} = -200 \text{ mV}$ and $E_{fin} = -900 \text{ mV}$ ($L_Q \approx 2.8 \cdot 10^{-7} \text{ mol} \cdot \text{L}^{-1}$ for 2,3-DMQ and $1.8 \cdot 10^{-7} \text{ mol} \cdot \text{L}^{-1}$ for 2-OH-3-MQ). The developed method was then applied to real samples of white wine, light beer and homemade cider.