

Electrochemical detection of diclofenac on ZnO-modified glassy carbon electrodes

<u>Katarina Aleksić</u>¹, Ivana Stojković Simatović², Ana Nastasić², Jovana Trajković², Smilja Marković¹

¹Institute of Technical Sciences of SASA, Belgrade, Serbia

²University of Belgrade – Faculty of Physical Chemistry, Belgrade, Serbia

Diclofenac (DCF) is widely used anti-inflammatory drug, which due to its stability and poor degradation in water represents chemical contaminant that can cause negative effects on the environment. Considering the use of DCF in pharmacology and its negative impact on the environment, the development of sensitive, selective, cheap, and fast, analytical devices is recently in focus of environmental scientists and engineers. It has been shown that electrochemical sensors (ECS) have advantages over traditional analytical methods for detection and monitoring of chemical pollutants in water. Crucial performances of ECS: selectivity, sensitivity, response time and portability, can be improved by modification of bare electrodes. Until now different materials have been used for electrodes modification, among them are noble metals, metal oxides, polymers and variety of carbonaceous materials [1]. We synthesized and characterized zinc oxide particles to modify the glassy carbon electrodes and to test them as sensors for electrochemical detection of DCF. Zinc oxide particles were synthesized by a glycine-nitrate combustion process using 1 M aqueous solution of zinc-nitrate hexahydrate and glycine in the molar ratio 6:5. Precursor mixture was heated on a magnetic stirrer, at first up to 80 °C in order to release excess water, then heating was continued to 170 °C, with dwell time of 2 hours, until slow flameless combustion occurred. In addition, to prepare highly crystalline particles, the obtained amorphous powder was calcined in air atmosphere, at 400 and 500 °C for 4 hours; prepared samples were denoted as ZnO-400 and ZnO-500, due to the calcination temperature. To determine phase composition and purity the prepared samples were characterized by XRD, Raman and FTIR spectroscopy, while field emission scanning electron microscopy (FESEM) was applied to determine particles morphology and size distribution.

Linear sweep voltammetry (LSV) was applied for electrochemical quantification of DCF using a three-electrode system that included a glassy carbon electrode as the working electrode, a saturated calomel electrode (SCE) as the reference electrode, and Pt foil as the counter electrode. The ink was prepared by mixing 10 mg of prepared particles, ZnO-400 and ZnO-500, and 1.5 mg carbon black with 40 μ L of 5 % Nafion solution, 225 μ L ethanol, and 225 μ L water. LSV is performed in 25 mL of phosphate buffer (0.1 M, pH 7.0) with the addition of diclofenac infusion solution (75 mg DCF / 3 mL, Galenika a.d.) in a portion of 1 μ L to completely 12 μ L. All measurements were done in a potential window of 0.2–1 V vs. SCE at a scan rate of 20 mV·s⁻¹.

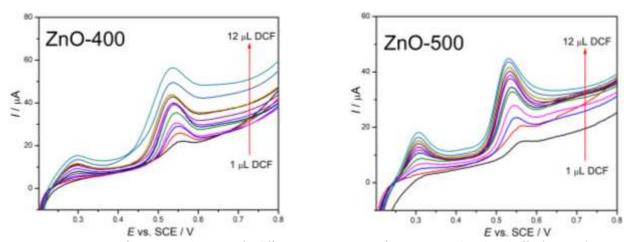


Figure 1. LSVs of ZnO-400 and ZnO-500 for different concentrations of DCF in 0.1 M Phosphate buffer (pH = 7.0)

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References

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