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Direct ultra-trace detection of alkylphenols in water using a cavity carbon-paste microelectrode sensor

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ABSTRACT

In this work, a new electrochemical method has been developed using a cavity carbon-paste microelectrode sensor (CP-CME) in cyclic voltammetry in order to detect alkylphenol micropollutants in water, especially 4-nonylphenol, 4-octylphenol and 4-*tert*-octylphenol. CP-CME sensor is characterized by small quantities of materials in the cavity and by a significant reduction of the electrochemical interface in comparison to the usual carbon paste electrode. Especially with the CME, the ohmic drop and the double layer capacitance are notably reduced in cyclic voltammetry allowing measurements of alkylphenol concentrations in water without any electrolyte addition. First, the electrochemical sensor was used for the detection of alkylphenols in a classical cell. Anodic oxidation of alkylphenols appears as an irreversible electrochemical process and experiments at different scan rates demonstrate that alkylphenol oxidation at the CME is not limited by diffusion but by a limited quantity of electroactive species (adsorption of alkylphenols). Also a surface activation step is required prior to a new measurement so as to liberate the adsorption sites occupied by oxidized alkylphenols and to subsequently adsorb alkylphenols. The developed method was validated by examining the linearity ranges, the repeatability, the accuracy, as well as the detection limits for each alkylphenol which are very low and ranged between 20 and 35 nM. The established calibration curves cover a large concentration range allowing alkylphenols quantification. The calculated accuracy accounts to 92 % and 99 % for the different alkylphenols, indicating that the developed method shows a good repeatability and very high accuracy. Furthermore, the electrochemical sensor was used in a special flow cell and alkylphenols could be also detected continuously by the CP-CME in adapted conditions. Flow rate and adsorption time were optimized in order to obtain good and repeatable signals. In conclusion, the CP-CME sensor has demonstrated its capability to detect alkylphenols continuously and directly in water.

Keywords: Cavity microelectrode sensor; Carbon paste; Cyclic voltammetry; Alkylphenols

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