Abstract

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Title of the diploma thesis: Spectrophotometric determination of chlorhexidine in mouthwash employing Lab-In-Syringe automated ion-pair extraction and back-extraction

In this work, a magnetic stirring-assisted dispersive liquid-liquid micro-extraction (MSA-DLLME) automated by the Lab-In-Syringe technique is presented. MSA-DLLME is based on mixing the sample and an immiscible solvent by the action of a magnetic stirrer for the dispersion of the solvent into fine droplet to enhance the extraction process. In Lab-In-Syringe, the magnetic stirrer is placed inside a syringe void, which is used as extraction chamber. To allow the use of an extraction solvent lighter than water, the syringe was turned upside down in this work. This method was designed for the determination of chlorhexidine in commercial mouthwash samples.

To allow the extraction of chlorhexidine, an ion-pair complex with the reagent methyl orange needed to be formed. After the extraction into an organic solvent, the aqueous solution was exchanged and the analyte was back-extracted into an acidic aqueous acceptor to yield higher selectivity. Spectrophotometric detection was used throughout.

Experimental parameters including type of extraction solvent, extraction times, volumes and, stirring rate were optimised. As extraction solvent, 1-octanol was chosen due to its highest extraction capacity of all tested solvents. Based on the experiments made, the extraction and back-extraction times were set to 30 s at a stirring rate of 1470 rpm.

Volumes of 1-octanol and methyl orange reagent were established at 250 µL and 50 µL, respectively. As back-extractant 500 µL of 0.125 mol/L hydrochloric acid was chosen.

The method performance was evaluated by the analysis of commercial mouthwash. Recovery values were below 100 %, requiring further studies on potential interferences.