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Environment and water

Optimization of SPE/HPLC analytical method for 17 β -estradiol quantification in wastewater treatment plant (in)effluents using a surface responsive methodology

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The ever-increasing use of endocrine disruptors compounds (EDCs), through pharmaceuticals such as synthetic estrogens, both in humans as well as in animals, are becoming ubiquitous in the environment. In addition to that fact, the poor removal by the conventional biological wastewater treatment plants, becomes a major source of their release into different aquatic matrices. Therefore, the occurrence and, more importantly, the destination of these compounds are matters of utmost importance towards a better public health. The present work is divided in two main experimental steps. First, an SPE/HPLC-UV1 experimental methodology is optimized to detect and quantify 17 β -Estradiol (E2) present into aqueous samples. The optimization of HPLC-UV operating conditions consisted of selecting a mobile phase composition that allowed a higher signal of E2, with a lower retention time, after analyzing 10 different compositions, methanol was the chosen one. In Figure 1, it is presented the chromatographic pulses of estradiol using 8 different concentrations (HPLC analysis without SPE extraction) in the 10 combinations of mobile phase tested. The solid phase extraction optimization comprises a three-level Box-Behnken (BBD) experimental design² with four factors to optimize (sample volume, sample pH, adsorbent drying time and solvent composition in the washing step), combined with a response surface methodology. After the SPE procedure was completed, the samples were analyzed in the HPLC-UV system. The highest responses were obtained with experiments that has pH2, that factor proved to be the one that contributed the most in obtaining a higher recuperation of E2. Figure 2 presents the influence that all 4 parameters, when modified, have in the response (chromatographic area). The validation of the optimized experimental methodology is done by the monitoring of estradiol in wastewater influent and effluent samples from a wastewater treatment plant.

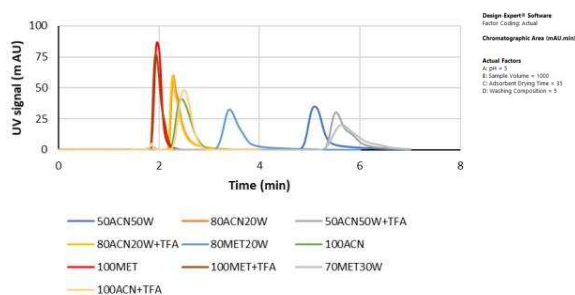


Figure 1. HPLC-UV chromatographic pulses of a E2 standard solution (100 mg/L) injected in 10 different mobile phase combinations.

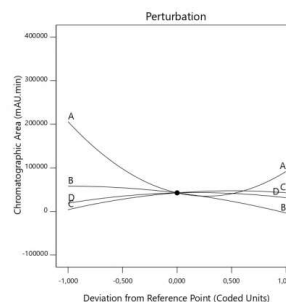


Figure 2. Perturbation graphic relating the influence of each parameter optimized with the response (chromatographic area).

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