OPTIMIZATION OF THE DISPERSIVE LIQUID-LIQUID MICROEXTRACTION METHOD FOR DETERMINATION OF BISPHENOLS AND HORMONES IN WASTEWATER

***Marco Tadeu Gomes Vianna*[1]**
***Rodrigo Coutinho*[1]**
***Marcia Marques*[1]**

[1)] *Department of Sanitary and Environmental Engineering. Rio de Janeiro State University - Brazil*

Abstract
The Dispersive Liquid-liquid Microextraction (DLLME) is a technique of extraction and concentration of compounds in aqueous matrices of high complexity, including effluent and environmental samples. The objective of this study was to find the best condition for a joint extraction/recovery for bisphenol A (BPA), bisphenol S (BPS), bisphenol F (BPF), bisphenol AF (BPAF), Benzophenone (BZP), 17α-Ethynylestradiol (17a-EE), 17β-Estradiol (17β-E) and estrone (EST) by DLLME using Design of Experiments (DoE) aiming at quantify these compounds in sewage samples from a wastewater treatment plant (WWTP) in Rio de Janeiro, Brazil. Analytes were identified and quantified using a Xevo TQD®, Quadrupole (UPLC-MS/MS Waters) equipment. The selection of variables and conditions considered relevant for DLLME optimization were performed using a Plackett-Burman Design (PB) followed by a Central Composite Design (CCD) for a joint optimization. After the optimization, the best extraction condition was applied in two real wastewater samples (raw and treated) from a WTTP located in Rio de Janeiro. The recoveries obtained with the raw and treated wastewater samples for all compounds of interest were within the range from 80 to 120%. The amounts of target analytes found in raw and treated wastewater samples were, respectively: BPA (150 ng mL^{-1}, 0.103 ng mL^{-1}), BPS (18.2 ng mL^{-1}, D-detected), BPF (0.091 ng mL^{-1}, D), BPAF (0.005 ng mL^{-1}, 0.016 ng mL^{-1}), BZP (0.923 ng mL^{-1}, 0.923 ng mL^{-1}), 17a-EE (1.17 ng mL^{-1}, 1.02 ng mL^{-1}), 17β-E (1.01 ng mL^{-1}, D), EST (1.10, D). The association between DLLME methodology and UPLC-MS/MS met the validation requirements adopted and enables high analytical standards. One of the additional advantages of applying this extraction technique is the low analytical cost per analysis (around $0.20) and the low volume of sample required, which makes possible to conduct chromatographic analyses of a large number of samples quickly, with simplicity and efficiency, making the entire process more sustainable.

Keywords: Dispersive Liquid-Liquid Microextraction, Hormones, Bisphenols, wastewater Design of Experiments, Optimization, Liquid Chromatography.

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