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Enantioselective Determination of Fluoxetine and Norfluoxetine in Wastewater by HPLC-FD using SPE

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Abstract: Microbial degradation of chiral compounds during wastewater treatment processes can be enantioselective and needs chiral analytical methodology to discriminate the biodegradation of both enantiomers. An enantioselective HPLC-FD method was developed and validated to monitor the degradation of fluoxetine (FLX) enantiomers by wastewater and the possible formation of its metabolite norfluoxetine (NFLX). The Solid Phase Extraction (SPE) of 50 mL of wastewater samples on 500 mg Oasis MCX cartridges was followed by the HPLC analysis using a Chirobiotic V chiral stationary phase under reversed mode. The developed method was validated within the wastewater effluent used in microcosms laboratory assays. The chiral SPE-HPLC-FD method demonstrated to be selective, linear, sensitive, accurate and precise to quantify the enantiomers of FLX and of its metabolites NFLX in wastewaters. The limits of detection ($0.8\text{-}2.0\text{ ng mL}^{-1}$) and quantification ($2.0\text{--}4.0\text{ ng mL}^{-1}$) were adequate to monitoring the degradation assays at environmental level. The method proved to be robust to follow the biodegradation assays using real wastewater samples spiked with FLX, during 46 days. To the best of our knowledge, this is the first report of simultaneous separation of FLX and NFLX enantiomers using a Chirobiotic V and the application of the validated method to the enantioselective degradation by wastewater.

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