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DI-SPME - On-fiber Derivatization - GC-MS. An innovative green and cost-effective approach to determine CECs and TPs from a novel anoxic-aerobic photobioreactor

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The demand of multicomponent methods for the analysis of compounds of emerging concern (CECs) in environmental matrices is a reality today. However, conventional techniques based on Solid Phase Extraction (SPE) coupled to Liquid Chromatography Mass Spectrometry (LC-MS) are very often only available in high-tech laboratories. The cost-competitive methodology presented here, successfully developed and validated, intends to fill the existing gap between current environmental needs and analytical capacities. It consists of an innovative method for the analysis of 12 CECs, including 3 Transformation Products (TPs), in sewage and sludge using a fully automatized online DI-SPME – On-fiber Derivatization – GC-MS. The validated method was proven to be reliable, thanks to the combination of two quantification approaches, i.e., matrix-matched and internal standard, as well as sensitive (LODs below 20 ng L-1 for most of the target compounds in sewage and 30 ng g-1 in sludge), versatile and green. The method was successfully applied to real samples from a novel pilot scale anoxic-aerobic photobioreactor, where the influence of the organic load on the removal efficiencies (REs) of the CECs was evaluated. The three operational stages, at three different concentrations of chemical oxygen demand (COD) (669±6 mg L-1, 493±11 mg L-1 and 434± 11 mg L-1), were maintained for 40 d (≈4 times the SRT) to achieve representative steady states. The maximum REs of ibuprofen, naproxen, salicylic acid, triclosan and propylparaben were 91±1%, 28±7%, 83±5%, 85±0% and 85±15%, respectively. COD concentration only affected clearly ibuprofen and naproxen REs. This pointed out oxidation as an important removal mechanism for those cases. In contrast, salicylic acid and triclosan REs slightly increased at lower COD loads. For propylparaben, high elimination rates (above 80%) were observed regardless the COD concentration. Oxidation, biodegradation, sorption, volatilization and photodegradation were discussed as the possible removal mechanisms of the tested contaminants. This constituted the first evaluation of CECs removal by a synergetic interaction between algae and bacteria depending on the organic carbon load.