Targeted screening by Orbitrap HRMS reveals the occurrence of pharmaceuticals, personal care products and pesticides in the Belgian Part of the North Sea

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In the last decades, extensive use of pharmaceuticals, personal care products and pesticides has led to an increasing concern about their environmental occurrence and impact. Poor removal by conventional wastewater treatment techniques has caused these emerging organic micropollutants to be ubiquitously detected in (treated) wastewater, riverine water, groundwater and drinking water. Yet, information concerning their presence in the marine environment is lacking because of, a.o., the often ultra-trace concentrations (≤ ppb levels). As a result, new monitoring approaches with suitable sample preparation procedures are of utmost importance to deal with the low concentrations in the marine environment. Furthermore, the use of modern high resolution mass spectrometry (HRMS) offers promising potential for multi-residue ultra-trace analysis of unknown (untargeted) and quantification of known (targeted) micropollutants.

This study focused on the targeted analysis of three compound groups – pharmaceuticals, personal care products and pesticides – in seawater samples originating from the Belgian Part of the North Sea (BPNS). A comparison was made between two solid-phase extraction methods followed by a newly developed UHPLC-Q-Orbitrap-HRMS method. Triplicate samples were collected during two sampling campaigns in winter 2016 and spring 2017, at 3 locations in the BPNS (harbour and open sea at Zeebrugge, harbour of Oostende). After pre-treatment, the samples were extracted in parallel on Bakerbond hydrophilic Speedisk® and on Oasis® HLB cartridges and separated through UHPLC on a Hypersil Gold column (50 mm x 2.1 mm, 1.9 µm) prior to full-scan HRMS analysis on a Q-ExactiveTM instrument.

Targeted screening resulted in the quantification of up to 51 out of 89 target compounds. Overall, higher concentrations were measured in winter 2016 as compared to spring 2017, with concentrations ranging from 0.15 to 370 ng.L-1 and from 0.13 to 210 ng.L-1, respectively. The most abundant micropollutants found were the non-steroidal anti-inflammatory drug diclofenac and the antiarrhytmic drug sotalol, having maximum concentrations of 370 ng.L-1 and 360 ng.L-1, respectively. Moreover, 6 out of 17 organic micropollutants included in the watch list for European Union water monitoring – defined in Decision 2015/495/EU – were retrieved in one or more samples from the BPNS, stressing the need for (inter)national regulations in order to protect our coastal and marine environment.

Acknowledgments

The authors like to acknowledge the Belgian Science Policy (BELSPO) for funding the NewSTHEPS project (BR/143/A2/NEWSTHEPS). The financial support from the Hercules Foundation (Flemish Government; AUGE/11/016) and from the Ghent University Special Research Fund (01B07512) is acknowledged for the UHPLC-Q-ExactiveTM and the automated SPE equipment, respectively.

Keywords: emerging organic micropollutants; Belgian Part of the North Sea; High Resolution Mass Spectrometry