Concentration Levels of Fluoroquinolones in Wastewater and Receiving Environment

Brief description of the dataset

This dataset comprises of weekly concentrations of fluoroquinolones and resistance *qnrS* gene in South West UK. The following fluoroquinolones were analysed: (±)-ofloxacin, (±)-ofloxacin-*N*-oxide, (±)-desmethyl-ofloxacin, (±)-lomefloxacin, (±)-moxifloxacin, *S,S*-moxifloxacin-*N*-sulfate, *R*-(+)-besifloxacin, (±)-prulifloxacin, (±)-ulifloxacin, (±)-flumequine and (±)-nadifloxacin; ciprofloxacin, desethylene-ciprofloxacin, norfloxacin and nalidixic acid.

Abbreviations:

n.d. – not detected
<MQL – below method quantification limit
Conc. -concentration
Influent – wastewater influent
Efflunet – wastewater effluent
River upstream – river water before treated wastewater discharge point
River downstream – river water after treated wastewater discharge point

Monitoring and analytical information

The study area and sampling points

Wastewater influent and effluent were collected for 7 consecutive days running from Wednesday to Tuesday between June and October 2015 from five major WWTPs contributing to one river catchment in the South-West UK and covering an area of approximately 2,000 km^2 and the population of ~1.5 million (this constitutes >75% of the overall population in the catchment). Influent wastewater samples were collected between screening and primary sedimentation. River water was collected from upstream and downstream of the effluent discharge point at varying distances depending on accessibility. River water was not collected for Site E as the WWTP discharges directly to the estuary.

Influent wastewater was collected as volume proportional 24 h composites with average sub-sample collection frequencies of approximately 15 minutes using an ISCO 3700 autosampler. Sub-samples (80 mL) were cooled to 4˚C (samplers were packed with ice) during collection to limit biological activity and pooled after 24 h (Petrie et al. 2016). Effluent wastewater samples were collected using time proportional approach due to the limited variation of this matrix over 15-minute intervals as discussed elsewhere. River waters (8 L) were collected as grab samples. All samples were transported to the laboratory on ice for further processing. Sample preparation and analysis

Antibiotic analysis using chiral liquid chromatography coupled with tandem mass spectrometry

Once in the laboratory, wastewater samples were filtered through GF/F 0.7 µm glass fibre filter (Whatman, UK) and 50 mL of filtered wastewater was spiked with 50 µL of a mixture of isotopically-labelled internal standards at 1 mg L^{-1}. Analytes were extracted using SPE and Oasis HLB cartridges (60 mg, Waters, UK), previously conditioned with 3 mL of methanol and equilibrated with 3 mL of ultrapure water. 50 mL of spiked environmental samples were loaded on HLB cartridges that were then washed with 1 mL of ultrapure water. The elution was carried out with 4 mL of methanol into 5 mL silanised glass tubes. The extracts were transferred to the TurboVap evaporator (Caliper, UK) and completely evaporated to dryness under nitrogen flow (5-10 psi). Samples were reconstituted with 0.5 mL of 10 mM ammonium formate/methanol 1:99 v/v with 0.05% formic acid and filtered through 0.2 µm PTFE filters. The filtered samples were transferred to polypropylene plastic vials bonded pre-slit

PTFE/Silicone septa (Waters, UK) and then 20 µL were directly injected into a chiral HPLC-MS/MS system. Samples were prepared and analysed in duplicate (Castrignano et al. 2018).

Samples were analysed using a Waters ACQUITY UPLC® system (Waters, Manchester, UK). Chromatographic separation of all the analytes was carried out using a chiral CHIRALCEL® OZ-RH column (5 µm particle size, L × I.D. 15 cm × 2.1 mm, Chiral Technologies, France) with a 2.0 mm × 2.0 mm guard filter (Chiral Technologies, France). The column temperature was set at 30°C. The autosampler was kept at 4°C. A mobile phase consisting of 10 mM ammonium formate/methanol 1:99 v/v with 0.05% formic acid was used at a flow rate of 0.1 mL min^{-1} under isocratic conditions.

The MS system was a triple quadrupole mass spectrometer (Xevo TQD, Waters, Manchester, UK) equipped with an electrospray ionisation source. Analyses were performed in positive mode with an optimised capillary voltage of 3 kV, source temperature of 350°C, desolvation temperature of 350°C and desolvation gas flow of 650 l h^{-1}. Nitrogen, supplied by a high purity nitrogen generator (Peak Scientific, UK), was used as a nebulising and desolvation gas. Argon (99.999%) was used as a collision gas. MassLynx 4.1 (Waters, UK) was used to control the Waters ACQUITY system and the Xevo TQD. Data processing was carried out on TargetLynx software (Waters, Manchester, UK).

The method was fully validated as described elsewhere (Castrignano et al. 2018).

Format of the dataset

Concentrations of quinolones in influent and effluent wastewater, and in the receiving environment during the monitoring week are in ng/l.

Discussion of results is provided in (Castrignano et al. 2020)

References

Castrignano, E., A. M. Kannan, E. J. Feil, and B. Kasprzyk-Hordern. 2018. "Enantioselective fractionation of fluoroquinolones in the aqueous environment using chiral liquid chromatography coupled with tandem mass spectrometry." *Chemosphere* 206:376-386. doi: 10.1016/j.chemosphere.2018.05.005.

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