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Innovative combination of QuEChERS extraction with on-line solid-phase extract purification and pre-concentration, followed by liquid chromatography-tandem mass spectrometry for the determination of non-steroidal anti-inflammatory drugs and their metabolites in sewage sludge

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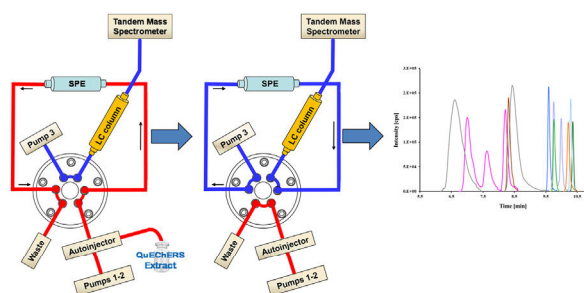
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HIGHLIGHTS

- Non-steroidal anti-inflammatory drugs and their metabolites are analysed in sludge.
- QuEChERS extract is automatically preconcentrated, purified and analysed by LC-MS.
- In most cases matrix effect was $\leq 20\%$ and recovery $\geq 50\%$.
- The determination of target analytes in sludge is achieved in 30 min.
- The method sensitivity is high, being it from tens of pg g^{-1} to ng g^{-1} of dry sludge.

GRAPHICAL ABSTRACT



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ABSTRACT

For the first time QuEChERS extraction of sewage sludge was combined with the automatic solid-phase pre-concentration and purification of the extract (following indicated as SPE) and LC-MS/MS analysis, for the determination of the non-steroidal anti-inflammatory drugs acetylsalicylic acid (ASA), diclofenac (DIC), fenbufen (FEN), flurbiprofen (FLU), ketoprofen (KET), ibuprofen (IBU) and naproxen (NAP), and their metabolites salicylic acid (SAL), 4'-hydroxydiclofenac (4'-HYDIC), 1-hydroxyibuprofen (1-HYIBU), 2-hydroxyibuprofen (2-HYIBU), 3-hydroxyibuprofen (3-HYIBU) and *o*-desmethylnaproxen (O-DMNAP). Various commercial pellicular stationary phases (i.e. silica gel functionalized with octadecyl, biphenyl, phenylhexyl and pentafluorophenyl groups) were preliminarily investigated for the resolution of target analytes and different sorbent phases (i.e. octyl or octadecyl functionalized silica gel and a polymeric phase functionalized with *N*-benzylpyrrolidone groups) were tested for the SPE phase. The optimized method involves the QuEChERS extraction of 1 g of freeze-dried sludge with 15 mL of water/acetonitrile 1/2 (v/v), the SPE of the extract with the *N*-benzylpyrrolidone polymeric phase and the water/acetonitrile gradient elution on the pentafluorophenyl stationary phase at room temperature. Matrix effect was always suppressive and in most cases low, being it $\leq 20\%$ for ASA, DIC, FLU, KET, IBU, 1-HYIBU, 2-HYIBU, 3-HYIBU, NAP and O-DMNAP, and included in the range of 35–47% for the other analytes. Recoveries were

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