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O Presidente da República

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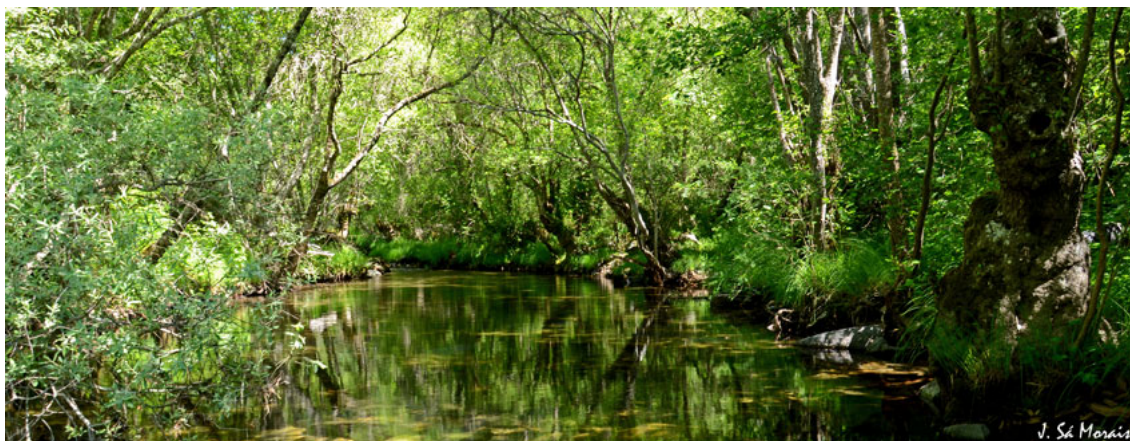
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PC-52

Coupling HPLC and GC-FID for the monitorization of oxidized intermediates from wet peroxide biphasic oxidation

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A GC-FID method capable to detect 2-nitrophenol (2-NP) and the resulting intermediates from its oxidation with H₂O₂ was developed and validated under the typical criteria for in-house pre-validation [1]. Linearity was demonstrated by F-test; accuracy and precision were assessed in three concentration levels. The oxidation process under study consists on the peroxidation of 2-NP, used as target lipophilic pollutant, in a biphasic mixture of cyclohexane(cC₆)/water to simulate contaminated oily streams. The oxidized intermediates produced by the peroxidation of 2-NP (P_{OW} = 51.3) in aqueous phase are phenol (Ph, P_{OW} = 30.2), hydroquinone (HQ, P_{OW} = 4.4), p-benzoquinone (BQ, P_{OW} = 1.9), catechol (CTL, P_{OW} = 7.6) and carboxylic acids [1]. Those compounds can be quantified in the aqueous phase by previous developed HPLC methods [2]. In the biphasic medium of reaction, the oxidation of cC₆ can also take place to produce cyclohexanone (cC₆O, P_{OW} = 5.8) and cyclohexanol (cC₆OH, P_{OW} = 21.9). The cC₆-water partition coefficient of 2-NP and the oxidized cyclic intermediates, *a priori* all lipophilic compounds, were assessed by analysis of the aqueous phase before and after addition of different volumes of cC₆. HQ and CTL keep their concentration in the aqueous phase after cC₆ addition and only the analysis of the other analytes (dissolved in cC₆) were done by GC-FID (Scion 436-GC, Bruker), without derivatization and adding a little quantity of Na₂SO₄ to remove the moisture. The injector and detector temperatures were set at 260 °C and 270°C, respectively. Separation was performed on a 50 m x 0.25 mm CP-Sil 88 column using the following temperature program: a first isotherm step at 160 °C, followed by a heating ramp at 5 °C/min, then at 10 °C/min and a final isotherm step at 220 °C during 5, 2, 5 and 5 min, respectively. At these conditions, the cC₆, cC₆O, cC₆OH, BQ, 2-NP and Ph compounds show well resolved peaks, and the method is capable to detect the compounds with maximum concentrations of 4.7, 2.3, 1.3, 1.0 and 7.1 g/L (in the same order) without overlap. The developed method, coupled with the HPLC methods, allows to follow the evolution of all compounds in the biphasic oxidation system.

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