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Rapid quantification of persulfate in aqueous systems using a modified HPLC unit

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ABSTRACT

Existing analytical techniques used for the quantification of persulfate (PS) in water mostly rely on polarography, reductometry or spectrophotometry. Although acceptable to a certain extent, these methods did not satisfy environmental chemists seeking rapid, reproducible and accurate quantification of PS upon the application of ISCO and AOPs technologies. Accordingly, a novel flow injection/spectroscopy analytical technique is developed via the use of an HPLC coupled to bypass capillary columns and a DAD detector. Special HPLC configuration uses concentrated KI solution as mobile phase to readily reduce PS present in the sample. The reaction takes place inside the capillary columns, under moderate pressure facilitating the production of Iodine suspension (I_2), to yield finally the formation of the Triiodide anion (I_3^-) in the presence of an excess of I^- . Triiodide absorbs at 352 nm which minimizes interferences from other organic contaminants (OCs). The method was validated by comparison to traditional PS quantification methods and tested on several environmental samples. The new method proved its superiority in terms of time requirement, labor need, material consumption, sample volume and simplicity. It eliminates the inconsistency present in other idiometric methods which is caused by the delay between the PS/I^- reaction and I_3^- measurement. The obtained LDR extends from 0.075 to 300 mmol L^{-1} with a LOD of $6.6 \times 10^{-3} \text{ mmol L}^{-1}$ and a LOQ of $2.20 \times 10^{-2} \text{ mmol L}^{-1}$. The method is successfully implemented in our laboratory to rapidly and automatically monitor the variation in the concentration of PS used in different projects, which facilitates the rapid determination of the reaction stoichiometric efficiency (RSE) of the oxidation reaction, a key factor toward the optimization of the mineralization process and its sustainability.

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