



Abstract New HPLC Method for Surfactants Detection in Wastewaters Samples ⁺

Iuliana Paun ^{1,2,*}, Laura Florentina Chiriac ¹, Florinela Pirvu ^{1,2}, Vasile Ion Iancu ¹, and Cristina Ileana Covaliu-Mierla ²

- ¹ National Research and Development Institute for Industrial Ecology—ECOIND, 57-73 Drumul Podu Dambovitei, 060652 Bucharest, Romania; laura.badea88@yahoo.com (L.F.C.); florinela_pirvu@yahoo.com (F.P.); vasileiancu10@gmail.com (V.I.I.)
- ² Faculty of Biotechnical Systems Engineering, University POLITEHNICA of Bucharest, 313 Splaiul Independentei, 060042 Bucharest, Romania; cristina_covaliu@yahoo.com
- * Correspondence: iuliana_paunita@yahoo.com
- Presented at the 1st International Electronic Conference on Processes: Processes System Innovation, 17–31 May 2022; Available online: https://ecp2022.sciforum.net.

Abstract: Over the last decade, biocides have received increasing attention due to their widespread use, their transfer to aquatic ecosystems, and their negative effects on aquatic organisms. Alkyl benzyl dimethyl ammonium chlorides are applied as bactericides and disinfectants in sanitary products and used as antistatic agents in the formula of laundry conditioners. The aim of this study was to provide a sensitive and robust HPLC-DAD method for the detection of three biocides (dodecyl- (C12-), tetradecyl- (C14-), and hexadecyl- (C16-) benzyl dimethylammonium chloride) in wastewater samples. The analytes separation was achieved using an Acclaim Surfactant Plus (3 μ m, 150 mm \times 3 mm) chromatographic column, maintained at 30 °C. The isocratic mode elution using a binary phase of ammonium acetate 0.2 M (A): acetonitrile (B) as mobile phase (50:50, v/v) at a flow rate of 0.5 mL/min, and allowed a run time of only 5 minutes. The linearity, accuracy, and intermediate precision were validated. The HPLC-DAD method provides good linearity, with correlation coefficients from 0.9992 to 0.9997 in the concentration range from 1 to 100 mg/L. Very good precision values were obtained, with RSD% ranged from 1.37-2.27% for intra-day measurements and between 6.14 and 6.65% for inter-day measurements. The target biocides were isolated from wastewater samples through the Solid Phase Extraction (SPE) procedure, using polymeric Strata-X Cartridges and acetonitrile and acetic acid (90%/10%) as elution solvent mixture. Recoveries (up to 86%) made possible the quantification biocides at very low levels, the limits of quantification (LOQs) were in the ranged between 4.5 and 7.6 μ g/L. The method was successfully applied to wastewater samples, obtaining concentration values varying from a few μ g/L to a few mg/L.

Keywords: HPLC-DAD; biocides; solid phase extraction; wastewater samples

Supplementary Materials: The following supporting information can be downloaded at: https://www.mdpi.com/article/10.3390/ECP2022-12622/s1.

Author Contributions: Conceptualization, I.P., L.F.C. and V.I.I.; data curation, I.P., L.F.C. and V.I.I.; formal analysis, I.P., L.F.C., V.I.I. and F.P.; investigation I.P., L.F.C., V.I.I., C.I.C.-M. and F.P.; methodology, I.P., L.F.C. and V.I.I.; project administration L.F.C.; resource L.F.C. and V.I.I.; supervision L.F.C. and C.I.C.-M.; validation L.F.C. and V.I.I., visualization, L.F.C., V.I.I. and F.P., writing—original draft, I.P. and L.F.C.; writing—review and editing F.P. and C.I.C.-M. All authors have read and agreed to the published version of the manuscript.

Funding: This research was funded by the Ministry of Research, Innovation and Digitization of Romania, The Romanian National Research Program "Nucleu" through contract no. 20N/2019, Project code PN 19 04 01 01.

Institutional Review Board Statement: Not applicable.



Citation: Paun, I.; Chiriac, L.F.; Pirvu, F.; Iancu, V.I.; Covaliu-Mierla, C.I. New HPLC Method for Surfactants Detection in Wastewaters Samples. *Eng. Proc.* 2022, *19*, 45. https:// doi.org/10.3390/ECP2022-12622

Academic Editor: Blaž Likozar

Published: 18 May 2022

Publisher's Note: MDPI stays neutral with regard to jurisdictional claims in published maps and institutional affiliations.



Copyright: © 2022 by the authors. Licensee MDPI, Basel, Switzerland. This article is an open access article distributed under the terms and conditions of the Creative Commons Attribution (CC BY) license (https:// creativecommons.org/licenses/by/ 4.0/). Informed Consent Statement: Not applicable.

Data Availability Statement: Not applicable.

Conflicts of Interest: The authors declare that there are no conflicts of interest regarding this publication.