SPICA 2022

18th International Symposium on Preparative and Industrial Chromatography and Allied Techniques

LISBON, PORTUGAL
OCTOBER 11-14, 2022
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BOOK OF ABSTRACTS

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ORAL COMMUNICATIONS : ABSTRACTS

FIXED-BED AND SIMULATED MOVING BED CHROMATOGRAPHY USING ACHIRAL AND CHIRAL ADSORBENTS FOR THE COMPLETE PREPARATIVE SEPARATION OF A QUATERNARY MIXTURE

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Nadolol is a nonselective beta-adrenergic receptor antagonist (beta-blocker) pharmaceutical drug, widely used in the treatment of cardiovascular diseases, such as hypertension, ischemic heart disease (angina pectoris), congestive heart failure and certain arrhythmias. This drug is still being marketed as a mixture of four stereoisomers in spite of the clear evidences that only one of its four stereoisomers has the desired therapeutic effect.

Preparative liquid chromatography is actually an accepted route in industry to obtain enantiomeric pure drugs. Today, the combining of different chromatographic technologies, such as fixed-bed and simulated moving bed together with the use of achiral and/or chiral adsorbents are an important improvement in performance of preparative separation of multicomponent chiral drugs into its single pure stereoisomers. This work confirms this improvement using the referred strategy with mobile phase composition optimization at both normal and reversed-phase mode. Different separation strategies were designed and optimized, enlarging the packing materials possibilities, from fully chiral (Chiralpak) to achiral (C18) combined with chiral separation. For each separation step, the optimization of the solvent composition was carried out with pure alcohol, alcohol-hydrocarbon and alcohol-water mixtures, all with a basic modifier (diethylamine), taking into account the strong basic nature of the nadolol.

Recently, our research group reported the pseudo-binary separation of nadolol by SMB chromatography using both coated Chiralpak AD and Chiralpak IA immobilized chiral stationary phases. In this work, we present an alternative strategy, implementing a first SMB achiral separation step, using six C18 preparative columns to perform the separation of the two pairs of nadolol racemates under reversed-phase mode. The extract and raffinate outlet streams of the SMB, both racemic mixtures, were collected and the solvent was removed to recover the two solid racemates. Then, new binary mixtures solutions were prepared and used for the design and optimization of two parallel binary chiral SMB separations. Results include an extensive set of experimental equilibrium and kinetic measurements, modelling, simulation and preparative separation. This methodology, developed during the last years by our group, will be explained and applied, to scale-up the separation process [1-4].

Acknowledgments:

This work was financially supported by: Base Funding - UIDB/00690/2020 of CIMO - Centro de Investigação de Montanha and Base Funding - UIDB/50020/2020 of the Associate Laboratory LSRE-LCM, both supports by national funds through FCT/MCTES (PIDDAC). This work is also a result of: Project “AIProcMat@N2020 - Advanced Industrial Processes and Materials for a Sustainable Northern Region of Portugal 2020”, with the reference NORTE-01-0145-FEDER-000006, supported by Norte Portugal Regional Operational Programme (NORTE 2020), under the Portugal 2020 Partnership Agreement, through the European Regional Development Fund (ERDF). Rami S. Arafah is supported by a PhD Grant of Fundação para a Ciência e a Tecnologia (SFRH/BD/137966/2018).

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