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Preparative Separation of Nadolol Racemates by Fixed-Bed and SMB Liquid Chromatography using C18 Columns

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Nadolol is one representative beta-blocker pharmaceutical drug prescribed worldwide for relieve of several diseases mainly related with the cardiovascular system. However, like other pharmaceutical drugs, it is also related with some severe risks, such as depression, insomnia and cardiovascular failure, among others. Some authors refer that this problem is related, or could be related, to the fact that nadolol drug is still marketed as a mixture of equal amounts of its four stereoisomers.

In the pharmaceutical industry and in terms of preparative scale, there are two main strategies to obtain pure enantiomer drugs. The first strategy is based on traditional methods of selective organic synthesis that allows the attainment of large amounts of a single product but with the disadvantage of being large time consuming. The other preparative strategy makes use of continuous separation processes based on multicolumn and cyclic adsorptive liquid chromatographic techniques to achieve the resolution of a racemic mixture. The use of such technologies allows, in a short period of time, the obtaining of enough amounts of pure enantiomers needed to perform the main pharmacological studies in the first steps of drug development.

Lately, our research group reported the pseudo-binary separation of RSR-nadolol stereoisomer, referred in the literature as being the one responsible of the nadolol drug therapeutic action, by means of simulated moving bed (SMB) chromatography using both coated Chiralpak AD and Chiralpak IA immobilized chiral stationary phases (CSP).^{1,2} This technology is generally based on the use of chiral adsorbents which must have enough recognition for all the chiral species.

In this work it is proposed an alternative strategy, implementing a first achiral separation step, to be followed by two subsequent parallel chiral separation steps.³⁻⁵ In this first achiral step, C18 columns are used to perform the separation of the two pairs of nadolol enantiomers ("racemate A" from "racemate B") under reversed-phase mode. The C18 achiral adsorbent allows the separation of the two pairs of nadolol diastereomers, i.e., the first racemate (composed by the nadolol compounds 2 and 3) co-eluting in the raffinate, and the second racemate (composed by the nadolol compounds 1 and 4) to be obtained in the extract SMB stream. After this preliminary achiral separation step, two parallel SMB runs must be carried out using a chiral stationary phase to achieve the complete separation of all the four nadolol stereoisomers.

Extensive experimental and simulation results will be presented including solvent screening, measurement of equilibrium adsorption isotherms, breakthrough measurements, and both fixed-bed (Azura prep LC unit) and SMB (FlexSMB-LSRE unit) preparative separations using C18 columns.

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