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Preparative Separation of Multicomponent Mixtures by Simulated Moving Bed Liquid Chromatography

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Direct racemic resolution of enantiomers by means of liquid chromatography using chiral stationary phases is nowadays a very popular technique. This popularity is mainly due to development of new chiral stationary phases and also by exploring and developing new and more efficient modes of operation. The use of chiral liquid chromatography through the simulated moving bed (SMB) technology has gained a renewed interest as an alternative technique for the production of fine chemicals and pharmaceuticals.

The classic SMB process is a continuous process to separate binary (or pseudo-binary) mixtures or to recover one single component from a multicomponent mixture. Several modified SMB processes have been introduced to separate multicomponent mixtures. Among them, the cascade SMB, the intermittent SMB, the JO processes and other complex multi-zone SMB related techniques, are often applied to the separation of multicomponent mixtures. The JO technology allows the separation of ternary mixtures through a cyclic process constituted by two discrete steps [1].

Nadolol is a pharmaceutical drug marketed as a mixture of four stereoisomers, used to treat cardiovascular diseases. However, its prescription is also related with some severe risks such as heart failure. It is well known that pure enantiomer separation is important to control chiral drugs safety. Recently, our research group reported the pseudo-binary separation of nadolol by SMB chromatography [2]. Using the classic SMB mode of operation, the complete separation of nadolol stereoisomers was achieved using Chiralpak AD chiral stationary phase (CSP). The more retained stereoisomer was collected 100% pure in the extract and a mixture of the other three stereoisomers was collected in the raffinate. In this work, we will present different strategies for multicomponent separation, using different solvent compositions, other CSP and SMB related techniques. Namely, (a) The use of Chiralpak IA, that comparing to AD CSP, allows the use of a wider range of solvents and therefore better separation performances; (b) The use of the JO process to achieve a final ternary separation, using the mixture of the three stereoisomers that co-eluted in the raffinate in the separation previously referred and (c) The separation of the two pairs of nadolol enantiomers using an achiral C18 material, followed by two parallel classic SMB binary enantioseparation processes.

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