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Amylose Derivatives as Versatile Chiral Selectors for Enantiomeric Separation in High-Performance Liquid Chromatography and Capillary Electrophoresis

Maher HM*

College of Pharmacy, Department of Pharmaceutical Chemistry, King Saud University, Riyadh 11495, P.O. Box: 22452, Saudi Arabia

Chiral molecules are constituents of a large proportion of therapeutic agents. Chiral compounds exist in two enantiomeric forms, which have identical molecular formula but they differ only in their interaction with the plane polarized light. The human body recognizes chirality and interacts differently with each enantiomer. Thus there can be marked differences between enantiomers in their pharmacological profile. Enantiomeric separations represent a critical subject in pharmaceutical analysis, since enantiomers of drug compounds may possess quite different pharmacological and toxicological properties. This is extremely important in order to obtain higher drug efficiency and to alleviate undesirable side effects. For this reason, establishment of rapid, selective and effective analytical methods has aroused a considerable need to verify the enantiomeric purity of chiral drugs [1].

In this context, HPLC columns with chiral stationary phases (CSPs) are gaining more interest. Among these, polysaccharide-based CSPs are very important in the field of chiral resolution.

Amylose is a well-known optically active polysaccharide which has an α-1,4-glycosidic linkage between its glucose units. These linkages result in groove formation with the fact that the backbone structure of amylose shows helical structure, unlike cellulose which has linear rigid polymers. Chemical derivatization of the native amylose backbone has been accomplished with an attempt to improve the chiral selectivity. Probably the most important adsorbing sites on phenylcarbamate derivatives are polar carbamate groups, which interact with racemic compounds via hydrogen bonding through –NH– and >C=O groups, and dipole–dipole interactions on >C=O [2]. Therefore, the nature of the substituents on the phenyl ring affects the polarity of phenyl groups, which results in different chiral recognition capacities. For this reason, many substituted benzoate and phenylcarbamate derivatives have been synthesized. The chiral recognition ability of amylose selectors depend primarily on differential interaction of enantiomers with the chiral cavities that arise from these helical formations. Moreover, the substituents on the phenyl group of derivatized amylose units play an important role in the chiral recognition. Di-substitution of the phenyl moiety of the derivatized amylose, with electron-donating groups (mainly methyl groups), with electron-withdrawing groups (halogens), or with both, affect the overall enantioselectivity. Moreover, the position of the substituent on the derivatized amylose backbone can affect the separation power dramatically. Also, tris (4-methylphenylcarbamate) and tris (5-chloro-2-methylphenylcarbamate) of amylose showed high chiral recognition. Among many amylose derivatives prepared so far, 3, 5-disubstituted derivatives such as 3, 5-dimethyl and 3, 5-dichlorophenylcarbamate showed particularly interesting and effective optical resolving abilities for a variety of racemic compounds. Derivatized amylose selectors have been used with different chromatographic techniques, such as normal-phase liquid chromatography (NPLC), reversed-phase liquid chromatography (RPLC) and polar organic solvent chromatography (POSC).

Although amylose based CSPs are gaining large reputation in the field of chiral HPLC separation, disadvantages of these methods however include the relatively expensive chiral columns, the large volumes of eluting mobile phases and the time consumption. Recently, applications of capillary electrophoresis (CE) in chiral separation have been increasing owing to its high resolution efficiency, the rapid method development, the short analysis times and the low consumption of reagents. The methods and research in capillary electrophoresis (CE) are one of the major areas of the chiral separation [3]. Since amylose is an excellent chiral selector, short chain analogues of native amylose have been used as chiral selectors in CE for a wide range of racemic drugs belonging to different pharmacological groups.

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***Corresponding author:** Maher HM, College of Pharmacy, Department of Pharmaceutical Chemistry, King Saud University, Riyadh 11495, P.O. Box: 22452, Saudi Arabia, Tel: 966-12914843; E-mail: hadirrona@yahoo.com

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