

Synthesis of a CSP and its application for preparative enantioseparation using open column chromatography

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Over 200 commercial CSPs are available in the market in the form of chiral HPLC columns and none of these has been used for *large scale separations in real terms*. The preparative HPLC is also not a large scale separation in real terms. There are no reports or attempts to develop a CSP that would be stable, less expensive and could be used by open column chromatography for preparative separation of enantiomeric mixtures from enantioselective synthesis or in pharmaceutical industries.This presentation reports synthesis and characterization of a simple chiral material by binding of BSA on the surface of normal silica gel and its application using simple open column chromatography for preparative enantioseparation in real quantitative terms by direct approach.

BSA bonded-silica CSP was developed as follows: (i) silica gel used for open column chromatography was treated with alkali that generated silanoxy group at the surface, (ii) treatment of alkali treated silica gel with 1,4-dibromobutane yielded 4-bromobutyloxysilica, (iii) the silica surface was further modified by treatment with diethylmalonate in presence of sodium methoxide, (iv) further, reaction with hydrazine hydrate produced carboxyhydrazide silica, (v) the carboxyhydrazide silica was reacted with BSA in presence of 4-Dimethylaminopyridine (DMAP) and DCC. The CSP was characterized by scanning electron microscopy (SEM), CHNS, FTIR, UV-Vis and XRD. The phase determination was done using XRD and the image obtained by it clearly revealed the highly amorphous structure of the silica surface corresponding to every step. The CSP successfully provided direct resolution of phenylalanine at a scale of 3.0 g with very good separation efficiency using simple open column chromatography. The CSP presented in this paper can be removed from the column, dried and then used on another occasion.

Biography

Prof. Ravi Bhushan worked on enantiomeric resolution at University of Oldenburg and Giessen in Germany on *Alexander von Humboldt fellowship*. He worked on primary structures of proteins of *Peanut*, *Brassica*, and *Soybean* and was awarded *European Economic Community Fellowship* in 1992 and worked at University of Bristol, UK, on *Rye* protein. He is an elected *Fellow of the Royal Society of Chemistry*, London, and *Fellow National Academy of Sciences India.* He is a member of the editorial board of *Biomedical Chromatography*, *Bioanalysis* and *Acta Chromatographica*. He presented research papers/plenary lectures at various International Conferences. He guided 32 doctoral and 53 Masters' theses, published more than 265 research papers and chapters in books and encyclopedia; edited four Special Issues of *Biomedical Chromatography* on *Chiral Resolutions* as Guest Editor. He founded ***IPR Cell*** in the year 2000; received the 'Outstanding Teacher Award' of the year 2007 of IIT Roorkee, He has expertise in enantiomeric resolution of compounds of pharmaceutical importance using LC.

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