Enantiomeric separation using temperature-responsive chiral polymers composed of -valine diamide derivatives in aqueous liquid chromatography

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„...value, which is the logarithm of the partition coefficient of the compound in octanol-water systems, was predicted using Pallas PrologP software (CompuDrug International) based on the structural formulas of the compounds. 3 Results..."

Abstract

This paper describes enantiomer separation by aqueous liquid chromatography using chiral stationary phases (CSPs) in which temperature-responsive polymers derived from acryloyl--valine *N*-methylamide (**1**) and its *N*,*N*-dimethylamide analogue (**2**) were bound on silica gel supports. The linear polymers composed of monomer **1** and monomer **2** are temperature-responsive in solution and their aggregation and extension states related to water solubility are reversible at particular critical temperatures. During chromatography, enantioselectivity and retentivity for solute enantiomers were controlled by column temperature, which changes the aggregation and extension states of the chiral polymers depending upon their interior hydrophobic nature. Two different types of CSPs were made: a temperature-responsive linear polymer derived from 3-mercaptopropyl silica gel, and another polymer cross-linked with ethylene dimethacrylate from 3-methacryloyloxypropyl silica gel. The former CSP could separate racemic *N*-(3,5-dinitrobenzoyl(DNB))amino acid isopropyl esters. Retention of the amino acid derivatives was prolonged with an increase in column temperature. Enantioselectivity was also enhanced with temperature increase until the particular critical temperature. The latter, cross-linked CSP could not provide enantioselectivity for the amino acid derivatives in aqueous media, although the chiral valine diamide moieties were effective for enantiomer separation in non-aqueous media. The degree of hydrophobicity and volume of the bonded phase formed by the polymers on the support surface was determined by measuring the fluorescence of pyrene.

Author Keywords: Amino acid diamide polymer; Amino acids; Acryloylvaline methylamide; Acryloylvaline dimethylamide

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