CYCLODEXTRIN NEWS

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Sulfobutylether Beta-Cyclodextrin as Chiral Selector in Capillary Electrophoresis

Cyclodextrins (CDs) are the most frequently used chiral selectors in the capillary electrophoresis [1]. For academic research usually single isomer CDs are used, but for practical purposes the random substituted CDs give often suitable results especially when CD derivatives produced industrially are used. Sulfobutylether beta-CD (SBE-β-CD) is a pharmaceutical excipient in US Pharmacopoeia as solubilizing agent [2] and is a component of several drug formulations in the market. Its composition is strictly regulated therefore the average degree of substitution (DS) and the distribution of the components of various DS fall always in a narrow range. The standard quality results in high reproducibility for analytical applications.

The first report on the application of SBE-β-CD in capillary electrophoresis (CE) was published in 1994 by the group of Valentino Stella [3]. In this pioneering work, the authors demonstrated that enhanced enantiomeric separation can be achieved when the electrophoretic mobility of the chiral selector is opposite to that of the analyte. The "extended" enantiorecognition ability of SBE-CDs, based on the advantage of the countercurrent flow of the negatively charged additive with respect to the electroosmotic flow (EOF), was then further deepened by other scientists. Along the years, the family of sulfobutylated CDs gained popularity and SBE-β-CD is often referred as the most versatile chiral additive among the negatively charged CDs (and CDs in general) as it allows the enantioseparation of a wide variety of pharmacologically active racemates.

The use of SBE-CDs in CE is suitable for the analysis of illicit amphetamine, methamphetamine, methcathinone and propoxyphene [4] and for the enantiomeric separation of ephedrine and related compounds [5]. Chankvetadze et al. described the use of SBE-β-CD as a chiral additive for the resolution of basic racemic drugs, such as clenbuterol, dimethindene, etilefrine, mefloquine and metomidate, in free capillary zone electrophoresis.

The resolution of the racemates was achieved with very low concentrations (micromolar) of the chiral additive and the high efficiency of the selector was attributed to its counter-current mobility in respect to the racemic solute [6].

Also Desiderio and Fanali reported on the effective use of SBE-β-CD as a chiral selector in CE in a study on the enantiomeric separation of warfarin, pindolol, propranolol, terbutaline, etc., a variety of underivatized anionic and cationic compounds of pharmaceutical interest as well as dansyl-amino acids [7].

SBE-β-CD was found as best chiral selector for the enantiomeric separation of several neutral and anionic herbicides by capillary zone electrophoresis [7]. SBE-γ-CD has been also investigated as chiral selector for CE. A comparative study between SBE-γ-CD and native γ-CD has shown that SBE-γ-CD is more efficient in the electrophoretic separation of cationic analytes, but it cannot generally replace γ-CD since the enantioselectivities of the two selectors are sometimes distinctly different [8]. Electrophoretic chiral separation of chloroquine and pemoline was effectively achieved by using 2.5 mM SBE-β-CD and 1.0 mM SBE-β-CD in 50 mM pH 2.5 sodium phosphate buffer, respectively [9]. Fundamental drugs used for the clinical treatment of Parkinson's disease, such as DOPA and structurally related compounds were resolved into their enantiomers by CE with SBE-β-CD as chiral selector [10–11]. The use of SBE-β-CD as chiral additive in CE was also useful for the separation of estrogens and amino acid derivatives [12–14]. The enantioseparations of the antimalarian erythro-mefloquine and its analogues were screened by CE using library of CD derivatives, including SBE-β-CD, as chiral selectors [15].

The chiral separation of the dihydropyridine calcium channel blockers (DHPs) with CE has been reported utilizing SBE-β-CD as the chiral selectors: it was found that the anionic SBE-β-CD gives a robust separation of amlodipine enantiomers when using acetonitrile - 20 mM NaH_2PO_4 (pH 3.95), containing 20 mM SBE-β-CD (35:100, v/v) [16]. Kong et al. developed an electrophoretic method for the enantioseparation of neutral m-nisoldipine, by comparing SBE-β-CD and carboxymethyl-β-CD (CM-β-CD) as chiral selectors. It was concluded that SBE-β-CD was superior chiral selector than CM-β-CD. It was also pointed out that the DS of the SBE-β-CD can remarkably influence the enantiorecognition process. In particular, high DS SBE-β-CD induced better investigated by CD-mediated CE and it was found that the enantiomers of the neutral dihydropyridines were baseline-separated only with SBE-β-CD [18].

Fillet et al. performed comparative chiral resolution studies among the commonly used neutral CDs with SBE-β-CD. Increased enantiomeric resolution for various acidic drugs was achieved by using dual systems comprising neutral β-CD and charged SBE-β-CD [19].

The extensive work on the use of anionic CDs in chiral CE have been summarized in excellent reviews [20–22]. De Boer et al. in their review reported several examples about the utility of sulfobutylated β- and γ-CDs as chiral selectors and the authors referred to SBE-β-CD

as the "most widely used anionic CD derivative" [23]. In the review by Mikuš et al. [24], some new achievements in the use of SBE-CDs are reported.

Further papers on the analytical application of SBE-CDs are summarized in Table 1.

Table 1. Examples of using SBE-β-CD as chiral selector

Analytes	References
tadalafil [a]	25
sitagliptin [b]	26
imperanene [c]	27
R-modafinil	28
ofloxacin and its metabolites	29
volinanserin and its intermediate	30
methylargininies	31
polycyclic aromatic hydrocarbons	32-34
bupivacaine hydrochloride enantiomers	35
isradipine enantiomers	36
chloroquine enantiomers	37
benzofurys, cathinones, diphenidines, ethylphenidate, methiopropamine and thiothinone	38
brompheniramine, chlorpheniramine, cetirizine and promethazine racemate [d]	39
pantoprazole enantiomers	40
retroamide enantiomers	41
alogliptin enantiomers	42, 43
trans-tramadol and its metabolites [e]	44, 45
pyroglutamic acid derivatives [e]	46

[a] SBE-α-CD, [b] SBE-β-CD (average DS ~4), [c] SBE-γ-CD (DS ~4), [d] SBE-β-CD and native β-CD dual CD system, [e] electrokinetic chromatography (EKC)

The versatility of SBE-CDs as capillary electrophoretic tools, together with their commercial availability largely justify the extensive research conducted so far on this family of derivatives. The large number and wide structural variety of guest molecules where efficient chiral resolution was achieved demonstrate not only the excellent enantiorecognition but also the general and high affinity complex forming ability of these cyclodextrin derivatives.

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Bibliography & Keywords of Selected Publications of the Month

Arima, H.; Motoyama, K.; Higashi, T.

Potential Use of Cyclodextrins as Drug Carriers and Active Pharmaceutical Ingredients

Review

Chemical & pharmaceutical bulletin, 2017, 65, 341-348;

Arima, H.; Motoyama, K.; Higashi, T.

Potential therapeutic application of dendrimer/cyclodextrin conjugates with targeting ligands as advanced carriers for gene and oligonucleotide drugs

Polyamidoamine dendrimer (G3) conjugates with α-CyD, Cell-specific drug carriers, Polyethylene glycol, galactose, lactose, mannose, fucose and folic acid-appended α-CDEs

Therapeutic Delivery, 2017, 8, 215-232; DOI:10.4155/tde-2016-0064

Shi, X.; Li, W.; Liu, H.; Yin, D.; Zhao, J.

β-Cyclodextrin induces the differentiation of resident cardiac stem cells to cardiomyocytes through autophagy

Regeneration of damaged hearts, Increased expression of cardiac transcription factors and structural proteins, Enhanced myogenic transition, Cholesterol efflux

Biochimica et Biophysica Acta, Molecular Cell Research, 2017, 1864, 1425-1434; DOI:10.1016/j.bbamcr.2017.05.012

Perez-Anes, A.; Szarpak-Jankowska, A.; Jary, D.; Auzely-Velty, R.

β-CD-Functionalized Microdevice for Rapid Capture and Release of Bacteria

Quartz-crystal microbalance, Functionalization of surfaces by β-CD, Selectively methylated β- CD derivative as a competitive host, Capture/elution strategy, Escherichia coli

ACS Applied Materials & Interfaces, 2017, 9, 13928-13938; DOI:10.1021/acsami.7b02194

Zhai, Z.; Zhang, F.; Chen, X.; Zhong, J.; Liu, G.; Tian, Y.; Huang, Q.

Uptake of silver nanoparticles by DHA-treated cancer cells examined by surface-enhanced Raman spectroscopy in a microfluidic chip

Cyclodextrin-coated silver nanoparticles modified with para-aminothiophenol and folic acid, Endocytosis, Targeted drug delivery, Controlled release

Lab on a Chip, 2017, 17, 1306-1313; DOI:10.1039/C7LC00053G

Sun, H-L.; Chen, Y.; Han, X.; Liu, Y.

Tunable Supramolecular Assembly and Photoswitchable Conversion of Cyclodextrin/Diphenylalanine-Based 1D and 2D Nanostructures

Supramolecular assembly of adamantanyl-modified diphenylalanine with azobenzene-bridged bis(β-cyclodextrin), Nanosheet, Nanotube, Photo-switchable one-dimensional/two-dimensional morphological interconversion

Angewandte Chemie, International Edition, 2017, Ahead of Print; DOI:10.1002/anie.201612629

Fine-Shamir, N.; Beig, A.; Zur, M.; Lindley, D.; Miller, J.; M. Dahan, A.

Toward Successful Cyclodextrin Based Solubility-Enabling Formulations for Oral Delivery of Lipophilic Drugs: Solubility-Permeability Trade-Off, Biorelevant Dissolution, and the Unstirred Water Layer

Success/failure of cyclodextrin-based solubility-enabling formulations for oral delivery of low-solubility drugs, Danazol, HPβCD, Excess CD

Molecular Pharmaceutics, 2017, Ahead of Print; DOI:10.1021/acs.molpharmaceut.7b00275

Oster, M. and Schlatter, G. and Gallet, S. and Baati, R. and Pollet, E. and Gaillard, C. and Averous, L. and Fajolles, C. and Hebraud, A.

The study of the pseudo-polyrotaxane architecture as a route for mild surface functionalization by click chemistry of poly(ε-caprolactone)-based electrospun fibers

Pseudo-polyrotaxanes of cyclodextrin and PCL, Core:shell fibers, Coaxial electrospinning, Grafting fluorescein isothiocyanate and bicyclononyne groups

Journal of Materials Chemistry B: Materials for Biology and Medicine, 2017, 5, 2181-2189; DOI:10.1039/C6TB03089K

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Thermoresponsive Self-Assembled β-Cyclodextrin-Modified Surface for Blood Purification

Removal of bilirubin from the body in clinics, Detoxification system, Adamantane on gold surface, β-CD dimer, Resistance to plasma proteins, In vitro platelet adhesion assay, Hemolysis assay, Negligible cytotoxicity

ACS Biomaterials Science & Engineering, 2017, Ahead of Print; DOI:10.1021/acsbiomaterials.7b00156

Benkovics, G.; Malanga, M.; Fenyvesi, E.

The 'Visualized' macrocycles: Chemistry and application of fluorophore tagged cyclodextrins

Review, chemosensing, Fluorescent microscopy, Synthetic strategies, Photodynamic therapy

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Template-directed synthesis of a cubic cyclodextrin polymer with aligned channels and enhanced drug payload

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Iron(II)porphyrin-Cyclodextrin Supramolecular Complex as a Carbon Monoxide-Depleting Agent in Living Organisms

Per-O-methylated β-cyclodextrin (TMe-β-CD), HemoCD

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Synthesis of multi-lactose-appended β-cyclodextrin and its cholesterol-lowering effects in Niemann-Pick type C disease-like HepG2 cells

Active targeting hepatocytes with β-CD, Decreasing the concentration of intracellular cholesterol, Negligible cytotoxicity, Increased internalization

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Dysregulation of Shh signaling, 2-Hydroxypropyl-β-cyclodextrin, Abnormal morphogenesis of the cerebellum of Npc1-deficient mice, Primary cilium

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HP-β-CD pro-drug, polyrotaxanes

Scientific reports, 2017, 7, 46737

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Synthesis and Characterization of Halloysite-Cyclodextrin Nanosponges for Enhanced Dyes Adsorption

Inorganic-organic nanosponge hybrids, Clay, Microwave irradiations in solvent-free conditions, Rhodamine B adsorption

ACS Sustainable Chemistry & Engineering, 2017, 5, 3346-3352; DOI:10.1021/acssuschemeng.6b03191

Rey-Rico, A.; Babicz, H.; Cucchiarini, M.; Madry, H.; Concheiro, A.; Alvarez-Lorenzo, C.

Supramolecular polypseudorotaxane gels for controlled delivery of rAAV vectors in human mesenchymal stem cells for regenerative medicine

Cartilage regeneration, Hyaluronic acid, Chondroitin sulfate, αCD, Improved transgene expression. Sustained release

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Supramolecular Polymeric Materials Containing Cyclodextrins

Review, Macroscopic self-assembly of polymer gels carrying host and guest residues, Stimuli-responsive self-healing properties, Macroscopic motion of artificial muscle

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Organophosphorus nerve agents, Competitive inclusion between the organophosphorus substrate and the iodosobenzoate group, Imidazole group

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Enantiomer migration order, Heptakis(2-O-methyl-3,6-di-O-sulfo)-β-CD, Shallow external complex

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Pressure inverse solubility and polymorphism of an edible γ-cyclodextrin-based metal-organic framework

***T**rigonal structure*

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Martel, B.; Campagne, C.; Behary, M. N.

When textiles help your recovery

Wound dressings, Implantable devices, Advanced spinning technologies of biostable or bioresorbable polymers and surface treatment technologies, Cyclodextrins releasing the active agents in situ

Medecine sciences : M/S, 2017, 33, 73-80

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Poly(carboxylic acid)-Cyclodextrin/Anionic Porphyrin Finished Fabrics as Photosensitizer Releasers for Antimicrobial Photodynamic Therapy

Polypropylene (PP) fabric finished with citrate-hydroxypropyl-βCD polymer, Photoinactivation of microorganisms, Staphylococcus aureus, Pseudomonas aeruginosa

Biomacromolecules, 2017, 18, 1134-1144; DOI:10.1021/acs.biomac.6b01752

Cheng, M.; Wang, M.; Zhang, S.; Liu, F.; Yang, Y.; Sun, L.; Wan, B.

Photocatalytic H2 production using a hybrid assembly of an [FeFe]-hydrogenase model and CdSe quantum dot linked through a thiolato-functionalized cyclodextrin

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