CYCLODEXTRIN NEWS

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Cyclodextrin Phosphates

In order to develop cyclodextrin derivatives having improved aqueous solubility, high solubilizing power, and low accumulation upon longer administration charged cyclodextrin derivatives are recommended. In addition to the sulfoalkylated and sulfated cyclodextrins the phosphated ones are also worth considering. In this editorial we summarize the literature on phosphate esters of CD monomers and polymers.

Phosphates of alpha-, beta- and gamma-cyclodextrin

The preparation is quite simple: by reacting a CD with a phosphorylating reagent such as phosphorus pentoxide or phosphorus oxychloride in the presence of an amide-based solvent or an ether-based solvent randomly phosphated CDs are obtained. [1] Using the reaction with phosphorous oxychloride the ratio of mono and diphosphate esters increased when the reaction temperature was raised from 25 to 60°C. [2] The monoesterified phosphate groups were mainly located at C6 of the anhydroglucose units when the reaction pH was 11 or 12. Reactions at pH 10, however, led to a higher degree of substitution at C2 than at C6. [2]

The synthesis of the selectively phosphorylated CD derivatives is more complicated:

- Alpha- and beta-CDs bearing one or two phosphate moieties on the primary rim were prepared by selective O-debenzylation of fully protected derivatives, followed by phosphorylation and deprotection. [3]
- Dialkyl chlorophosphates were used as phosphorylating agents for the synthesis of BCD derivatives bearing only one phosphate group on the primary rim. [4] These monosubstituted compounds were prepared in good to excellent yields in the presence of 4-dimethyl amino pyridine catalyst and dimethylformamide as solvent. The methodology described is highly selective and the purification is simple, because difficulties due to mixture of phosphate regioisomers in the randomly phosphated CDs are avoided.

Random phosphorylation results in enhanced solubility of CDs, especially of BCD, both in water and in ethanol:water (1:3) mixture. [2] The improved solubility is accompanied by enhanced solubilizing effect as shown in Fig. 1. The solubility isotherms of four model drugs in solutions of phosphate esters of BCD and GCD are compared to those of HPBCD. As it was expected the solubilizing effect depends not only on the cavity size of the CD phosphate but also on the chemical structure of the guest molecule. The solubilizing capacity of both phosphate esters surpasses that of HPBCD in the case of furosemide, while they underperform compared to HPBCD for hydrocortisone. The GCD phosphate was the best solubilizer among these 3 CD derivatives for the large molecules of amphotericine B, while the BCD phosphate was the best for the smaller ibuprofen.

Even better results can be obtained for the basic drugs. For instance, 7.8 mg/mL concentration of the practically water-insoluble (< 0.1 mg/mL) mebendazole can be achieved in 10% BCD phosphate solution. [1] Cyclodextrins bearing pendant cationic or anionic moieties have been shown to form highly stable inclusion complexes with oppositely charged organic molecules. [5]

The inclusion complexes of anionic CDs, such as CD phosphates with cationic guest molecules, such as vatalanib succinate, and the nanoparticles comprising these inclusion complexes are disclosed in a patent of Schering published in 2007. [6]

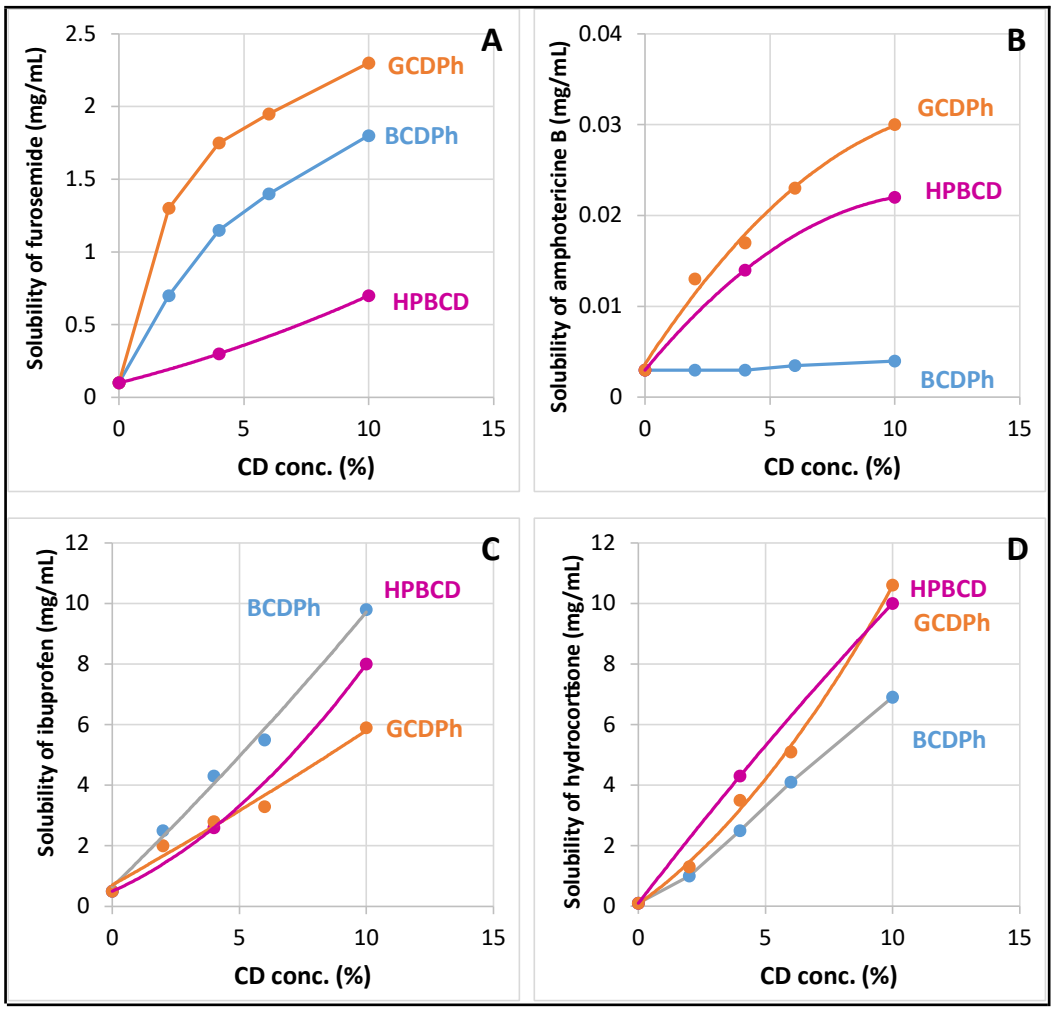

Fig. 1 Solubility isotherms of model drugs in aqueous solutions of phosphate esters of BCD and GCD (BCDPh and GCDPh, resp.) of DS 4 and of HPBCD [7]

CD phosphates can be used on various fields, including pharmaceutical industry, analytical applications and other industrial procedures.

Concerning the pharmaceutical applications, CD phosphates are not only good solubilizers, but they have a therapeutic effect: similarly to CD sulfates, they have anti-retroviral activity. [8] CD phosphates showed anti-HIV activity as high as that of CD sulfates, but their anti-coagulant activity was even lower than that of CD sulfates. [9]

Lisozyme refolding with CDs was found to depend on the ability of CD to suppress aggregation of the protein. [10] The presence of anionic substituents like phosphate groups promoted aggregate formation, and this way reduced the refolding ability compared to neutral CD.

The presence of both anionic and cationic substituents on the same CD molecule was found to partially restore its renaturation ability.

CD phosphates, similarly to other charged CDs are useful as chiral selectors for enantioseparation of hydrophobic drugs, including corticosteroids, such as triamcinolone acetate by capillary electrophoresis. [11] Metropolol enantiomers were successfully separated (Rs 2.7) by using alpha-CD phosphate in the background electrolyte. [12] Phosphated-gamma-CD provided lower detection limits, better repeatability of peak areas and migration times than sulfated alpha- and beta-CD for separation of R,S-tolterodine and R,S-methoxytolterodine enantiomers. [13] Phosphated-gamma-CD was the most appropriate for the simultaneous chiral determination of venlafaxine, an antidepressant drug and its main active metabolite, *O*-desmethyl venlafaxine in clinical samples. [14] Mono(6-O-diphenoxyphosphoryl)-beta-cyclodextrin (I) and mono(6-O-ethoxyhydroxyphosphoryl)-beta-cyclodextrin are chiral selctors

in amino acid analysis [15]. In another study, CD phosphates showed lower affinity for the analytes compared to CD sulfates in enantioseparation of some chiral drugs. [16] Anyhow, CD phosphates increase the choice of the CD derivatives useful as chiral selectors.

A few industrial applications of CD phosphates were also described:

- Cu salt of BCD polyphosphate was used as catalyst in vinyl polymerization with increasing catalytic activity as the number of phosphate groups on the CD increased. [17] The catalytic effect of the methylated CD/Cu complexes was also improved by introducing a phosphate group into the methylated CD. [18]
- CD phosphate was suitable as template for the preparation of organogels with a large variety of planar guests, like polyaromatic hydrocarbons, such as chrysene, using DMF, pyridine, or other polar solvents. [19]
- Similarly to other CDs, phosphated beta-CDs are degraded on heating in inert atmosphere in one major step (252-400°C) leaving a residue (char) which is thermally quite stable. [20] On heating in air, however, unlikely to other CDs where the char is oxidized to volatile products below 600°C, the phosphate substituted cyclodextrins give a ceramic-like residue stable to above 800°C. Cyclodextrin phosphates as flame retardants were described by Wada et al. [21]

Phosphates of alpha-, beta- and gamma-cyclodextrin polymers

The synthesis of β-CD polymer phosphate sodium salts was worked out to be used for coating of metal-organic framework (MOF) nanoparticles. [22] Epichlorohydrin was used as crosslinking agent and phosphorus pentoxide was applied for introducing phosphate groups (Fig.2). The phosphorylated CD polymers have high affinity to iron atoms within nanoMOF resulting in 26% weight increase caused by the coating shell (compared to 20% with the unphosphorylated CD polymer). The grafted phosphate moieties enabled a firm anchorage of the coating to the nanoMOFs. Coating stability was directly related to the density of grafted phosphate groups, and did not alter nanoMOFs morphology or drug release kinetics.

Phosphated beta-CD polymers were prepared by crosslinking BCD and BCD/dextran mixture with sodium trimetaphosphate, a non-toxic cyclic triphosphate. [23, 24] The resulting soluble CD polymer phosphates with molecular weights (Mw) higher than 10^4 g.mol^{-1} can form inclusion complexes, and show strong interactions with a divalent cation, Ca^{2+}. The strong affinity between these novel phosphorus-containing cyclodextrin polymers and hydroxyapatite was demonstrated by adsorption studies. Polymers that combine cyclodextrin and phosphorus functionalities is suitable for biomedical applications that jointly require calcium affinity and delivery of lipophilic bioactive molecules.

Beta-CD and β-glycerophosphate were crosslinked with hexamethylenediisocyanate to obtain cyclodextrin polyurethanes containing phosphate groups. [25] The CD cavities within this polymer preserved the ability for complex formation and showed controlled release of ciprofloxacin, as model drug.

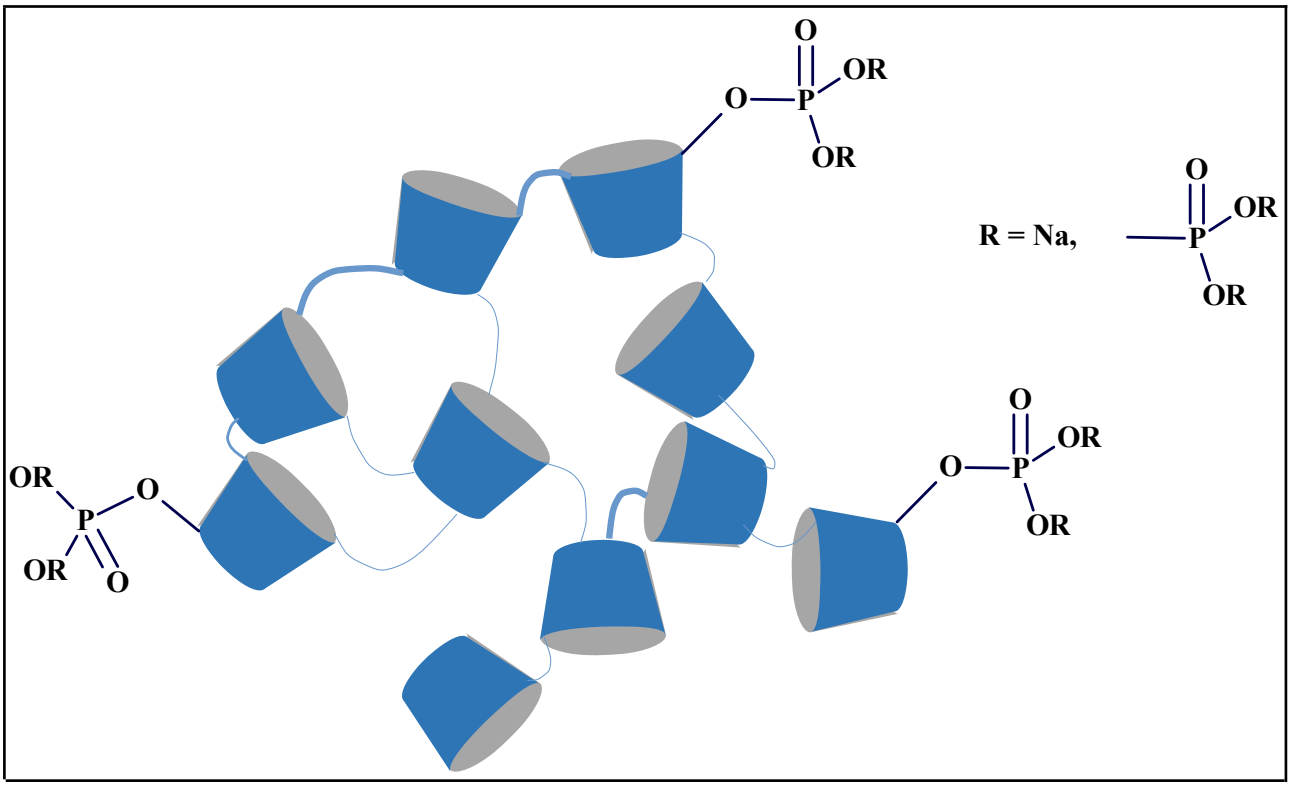

Fig. 2 Scheme of CD polymer modified with phosphate groups

A polyionic derivative of an alpha-, beta-, or gamma-CD polymer (water-insoluble) in which the ionic group is sulfate, phosphate, carboxylate, or nitrate was patented by Weisz et al. These ionic derivatives of CD polymers were found useful for purification of proteins, such as heparin-binding growth factor, from mixtures. [26]

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Cyclodextrin News Retrospective

We wrote 10, 20 and 30 years ago

10 years ago, CD News reported on some theme-selected works presented at the Kyoto Cyclodextrin Symposium held between 8-11 May 2008. The editorial dealt with the use of CDs in nanotechnology, highlighting the following sub-topics:

- Nanoparticles for drug delivery
- Nanoassemblies for gene delivery
- Nanotubes including carbon nanotubes
- Supramolecular structures, nanowires
- Nanoparticles in catalysis
- Nanocoatings on surfaces

This versatile group of publications consisted of 45 presentations (total number: 194), which means that almost every 4th presented themes were dealing with dispersion or association colloids as well as interfaces. A small section of CD News 22(6), 2008 announced the following historical information which was a milestone in cyclodextrin chemistry as seen from 10-year perspective:
„On 30 May 2008 the Committee for Medicinal Products for Human Use (CHMP) adopted a positive opinion, recommending to grant a marketing authorisation for the medicinal product Bridion 100 mg/ml, solution for injection intended for the reversal of neuromuscular blockade induced by rocuronium or vecuronium. The applicant for this medicinal product is N. V. Organon."

20 years ago, the editorial was a dissemination of the beta-cyclodextrin monograph in the European Pharmacopoeia, as it was included shortly before (1997) in the third edition.

30 years ago, a surprisingly futuristic theme was discussed on the front page of CD News: cyclodextrins and light-energy. The highlights of this editorial were:

- Solar energy generation by utilizing finely dispersed Pt and Rh particulates, wherein the adsorbed CD molecules provide long-term colloid stability
- Solar energy storage technology wherein hydrogen is generated from water in a CD-assisted process simulating photosynthesis
- Laser dyes
- Photochromics
- Reprographics
- (Classical) photographic applications

Probably, only the last item may be considered outdated (but still prescious from retrospective point of view), the rest are hot topics even today. Solar energy storage is a still unresolved challenge that is a missing link to utilize solar power for truly competitive 24-hour electricity generation. As photovoltaic power soon becomes the world's largest source of electricity, the importance of storage is emerging. Who knows, maybe CDs will also be used for the „controlled release" of electric power in the future?

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Selective attachment of dendrons in the seven positions of the βCD-primary face, CuAAC click reaction, Albendazole

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Vorinostat, HPBCD, PEG, Delayed neurodegeneration, Macrophages

Scientific Reports 2018, 8(1),3875

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Reactive oxygen species-mediated mitochondrial apoptosis, Suppressed tumor growth

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DOI:https://doi.org/10.1016/j.ejps.2017.10.039

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Development of a non-toxic and non-denaturing formulation process for encapsulation of SDF-1α into PLGA/PEG-PLGA nanoparticles to achieve sustained release

Trapping cancer cells, Stromal cell-derived factor-1α (SDF-1α), Poly-(lactic-co-glycolic acid) (PLGA), Polyethylene glycol (PEG)-PLGA co-polymer, HPBCD as stabilizer, Sustained release, Negligible protein denaturation

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CD aggregate formation, Solid dosage forms, Parenteral formulations, Ocular, Nasal and pulmonary administration

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9, 12, and 18 arms, Ring-opening polymerization, Atom transfer radical polymerization, Poly(ε-caprolactone), End-capping with ferrocene, Poly(oligo ethylene glycol) methacrylates, BCD, Doxorubicin, Oxidation-induced dissociation of BCD/ferrocene pair

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Chapter 10 - Getting under the skin: Cyclodextrin inclusion for the controlled delivery of active substances to the dermis

Cosmetoceuticals, Dermocosmetics, Dermotherapeutics, Skin penetration enhancing effect

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Grafting alkyl chains on the primary and/or secondary face, Co-nanoprecipitation, Polyethylene glycol (PEG), Healthy mice, In vivo elimination

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Multilayers, Poly-l-lysine, Poly-l-glutamic acid, Carboxymethyl-BCD, Photoactivated antimicrobial surfaces

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Lipofectamine, Amphiphilic polycationic cyclodextrin, Targeted nanoparticles, Adamantyl-PEG-ligands, Cationic polymer chitosan

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Chapter 15 - Cyclodextrin-based nanoparticles

Cyclosert delivery platform, Rondel platform, Gene delivery

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Conjugated heptapeptide, Hydrophobic interactions

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Dipalmitoyl phosphatidyl choline (DPPC), Saturated and unsaturated phospholipids, Cholesterol, HPBCD

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Prebiotics, ACD, Activating colonic microbiota

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14 - Authorised EU health claim for alpha-cyclodextrin

Reduction of post-prandial glycaemic responses, Foods, Nutrients and Food Ingredients with Authorised EU Health Claims

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CD as a carrier matrix and a feeding source for the encapsulated bacteria, Treatment of heavy metals and textile dye

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Energy conversion and storage processes, Molybdenum-based catalysts, Cyclodextrin as structural guiding agent and carbon source

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