

Highly Ordered Architectures Built from Cyclodextrin Complexes

Finding explanation for existence of order in nature has been a major scientific inspiration throughout thousands of years dating back to Antiquity. Every one of us may experience that our world is abundant in patterns (observable regularities). These patterns including symmetries, helices, waves, layers, etc... all may be manifested in various physical characteristics and can sometimes be modeled mathematically. Early Greek philosophers (e.g. Plato [1], Aristotle [2] Pythagoras [3] and Empedocles [4]) studied patterns attempting to explain presence of order in our world.

In the Middle Age Fibonacci adapted general mathematical model to describe periodicity in nature [5].

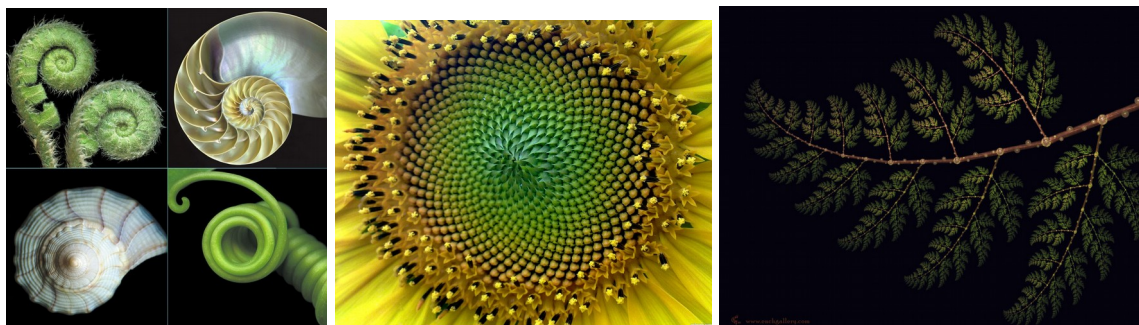


Figure 1. Examples of natural macroscopic ordered structures

In chemistry, systematic structural analysis of ordered substances has begun in the late 19th century with the revolutionary discovery of X-rays by Wilhelm Conrad Röntgen in 1895. Max von Laue investigated the interaction of X-rays with crystals producing a diffraction pattern. In 1913 the thorough research activity of William Henry Bragg and his son, William Lawrence Bragg refined the discipline of X-ray crystallography. Building on the work by von Laue, they formulated the relationship between a crystal's atomic structure and its X-ray diffraction pattern providing a tool which has revolutionized our understanding of the structure of matter ranging from minerals, pharmaceutical materials and catalysts to DNA, proteins and viruses.

A peculiar ordered state of matter, *liquid crystals* were discovered by Friedrich Richard Reinitzer performing experiments with cholesteryl benzoate in 1888. Otto Lehmann studied Reinitzer's peculiar material with polarized microscopy invented by himself. Later, Lehmann coined the term „fließende Krystalle“ (liquid crystal) in 1889.

In modern science, Pierre-Gilles de Gennes Nobel Prize laureate in physics (1991) became renown for methods developed for studying order phenomena in simple systems which can be generalized to more complex forms of matter, in particular to liquid crystals and polymers.

Some specially designed cyclodextrin derivatives are thermotropic liquid crystals in themselves, i.e. ordered liquid crystalline phases occur in certain temperature ranges. Derivatives of this property (heptakis(6-S-alkyl-6-thio)- β -CDs - where alkyl is a C2, C4, C10, C16 and C18 unit) forming thermotropic liquid crystals were first reported by Ling et al. [6]. Yang et al. synthesized three cyclodextrin-triphenylene derivatives by introducing the triphenylene unit on cyclodextrin by click chemistry and further acylation of hydroxyl groups of the cyclodextrin unit [7]. One of the three studied CD-based compounds showed column liquid crystal behavior: molecules assemble into elongated, cylindrical structures. Chen et al. constructed a well-defined structure liquid crystal built up of heptakis [6-deoxy-6-(1-H-1,2,3-triazol-4-yl)(methyl)6-(4-methoxybiphenyl-4-yloxy) hexanoyl]- β -cyclodextrin (H6B- β -CD) [8]. Differential scanning calorimetry (DSC), polarizing optical microscopy (POM), and wide-angle X-ray diffraction (WAXD) measurements showed that the supramolecular structure of H6B- β -CD is consisted of a large scale ordered lamellar structure and a small scale ordered structure at low temperature region. By increasing temperature, the small scale structure becomes disordered relatively in the first instance, from smectic E to smectic A phase. Then, the lamellar structure collapses and nematic phase then isotropic phase are observed in sequence.

Albeit relatively rare phenomenon, some cyclodextrin complexes may show liquid crystalline properties in aqueous media. Sony corporation designed a thermotropic liquid crystal system comprising liquid crystal materials in widespread use in TFT (thin-film-transistor) screens wherein cyclodextrin (permethylated beta cyclodextrin) is present as additive. In the transistors, inclusion complex is formed, the viscosity is lowered, consequently the response time is reduced [9].

Unlike the previous example, most of the CD-complexes showing liquid crystalline properties are lyotropic, i.e. the associates comprise hydrophilic and hydrophobic domains which may aggregate alongside and the resulting solvent-induced extended anisotropic arrangement generates the long-range order of the phases. Szejtli and Gerlőczy documented liquid crystalline amiodarone.HCl / γ -CD complex in 1993 (the results were in fact not published by the observers) when they attempted to record solubility isotherms of the drug [10]. Amiodarone.HCl in aqueous solution comprising γ -CD showed different phases in concentration dependent manner at room temperature:

- | | |
|-------------------------|---|
| Below 1 % γ -CD: | regular drug solubilization |
| 1 – 4 % γ -CD: | three phases (precipitate, „pearly gel-like” substance + supernatant) |
| 4 – 8 % γ -CD: | only solid phase + „pearly gel-like” phase |
| 8 – 10 % γ -CD: | only „pearly gel-like” phase (see Figure 2 for appearance) |



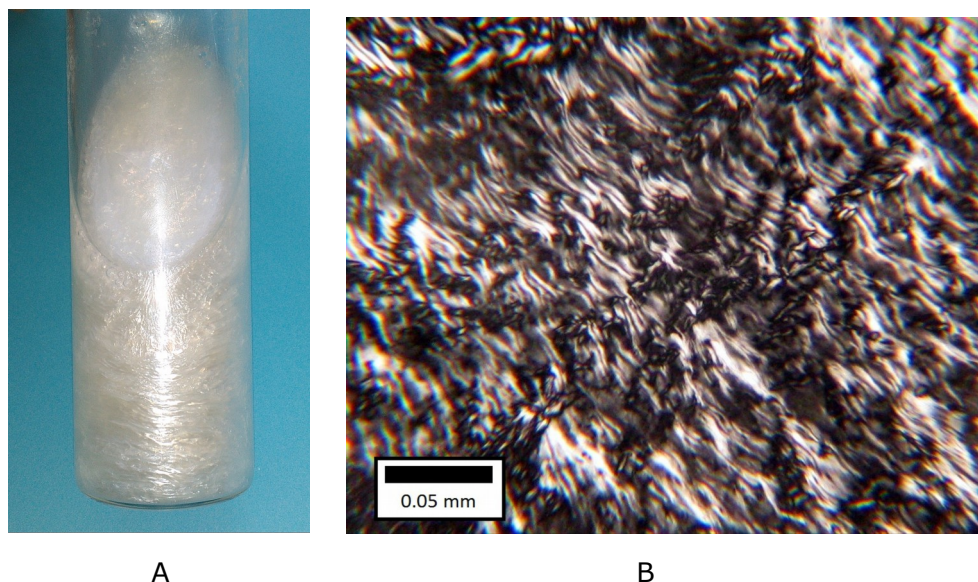


Figure 2. A) Appearance B) Polarized microscopic image of „pearly gel-like” phase (8% amiodarone.HCl / γ -CD complex) at room temperature

The complex in aqueous phase (8% amiodarone.HCl / γ -CD) shows peculiar phase transitions by increasing temperature. The gel phase observable at room temperature collapses at approximately 54 °C. This change can also be observed by DSC: an endothermic enthalpy change may be recorded. Above this temperature, the anisometry in structure is still visually detectable. Structural, hence optical inhomogeneity (as observed from different directions) caused by the orientation of the layers results in so-called Schlieren effect upon stirring (see spectacular texture in Figure 2A). At 60 °C thixotropic liquid crystal phase is present, while at 90 °C the liquid becomes anisometric, a sol phase forms.

Another drug substance, cinacalcet forms crystalline complex (precipitate) with γ -CD. If the solid complex is re-wetted with water, distinct Schlieren texture is obtained, in other words, liquid crystalline-like mesophase is formed [11]. Figure 3 shows the appearance of 1:2 mol/mol cinacalcet complexes under light microscope: liquid crystalline γ -CD complex versus a conventional, anisometric, amorphous cinacalcet/HPBCD complex is depicted.

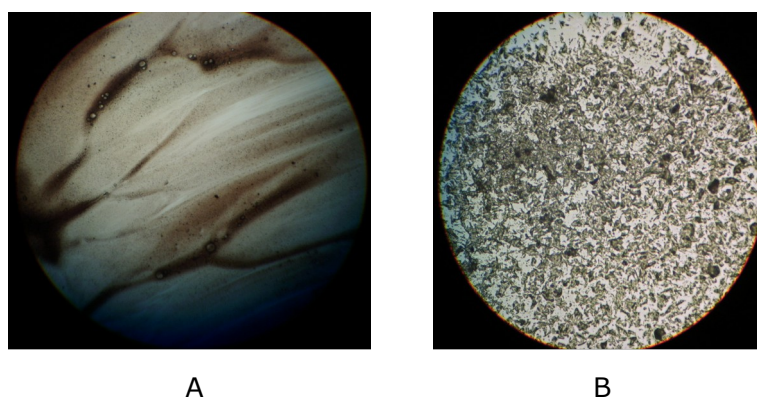


Figure 3. Appearance of 1:2 mol/mol cinacalcet complexes under normal light microscope A) prepared with γ -CD, B) prepared with HPBCD



Besides the scientific peculiarity of this structural arrangement, this phenomenon in fact may be exploited for practical use. It is notable that although the binding constant of cinacalcet and γ -CD is fairly low (48 M^{-1}) compared to that recorded with HPBCD (194 M^{-1}), however upon the reconstitution of the γ -CD construct, almost two times higher cinacalcet concentration could be attained compared to the HPBCD complex. The explanation of this apparent contradiction is thought to be that the liquid became supersaturated when the liquid crystalline phase was present.

Similarly, tapentadol forms lyotropic liquid crystalline complex with γ -CD, whereas, α -CD and β -CD forms anisotropic complex [12].

A number of surfactant compounds may also form liquid crystalline compositions with different cyclodextrins [9]. Some representative examples are listed below:

- Sodium dodecyl sulfate / α -CD
- Benzoxonium chloride / α -CD
- Benzalkonium chloride / α -CD, γ -CD
- Cetylpyridinium chloride / α -CD, γ -CD
- Cetyltrimethylammonium bromide / α -CD, γ -CD

Using cyclodextrin, more complicated ternary ordered structures may also be constructed e.g. using cationic surfactants, γ -CD and natural anionic polymer hyaluronan as building blocks [13]. The ordered structure of this ternary system can be controlled by modulating electrostatic interactions and host-guest inclusion phenomena. This ternary construct was proposed to be used as tunable drug delivery vehicle. A similar approach was applied for formulating anti-HIV drugs: Song et al. prepared supramolecular assembly containing hyaluronic acid, cationic polymer (poly-L-lysine) and anionic sulfobutylether- β -cyclodextrin [14]. HIV reverse transcriptase inhibitors zidovudine and lamivudine were successfully encapsulated into the polymer assembly in a noncovalent manner. The physicochemical properties and antiviral activity of the polymer assemblies were also studied. The results of this study suggest that the supramolecular assemblies loaded with HIV drugs exert potent antiviral activity and allow sustained drug release.

The above examples illustrate the versatility of ordered structures in the world of cyclodextrins. It is hoped that this editorial highlights that periodicity in the setting-up of these constructs is not only splendid and visually attractive but also may possess true practical relevance.



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Silica carrier, β -Cyclodextrin, Bioavailability

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Poly(N-isopropylacrylamide), Anticancer cytotoxicity, Doxorubicin

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Review, Neurodegenerative disease, Cyclodextrins, Fibrillation of different proteins/peptides

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Antitumor, Antigen-mediated cellular uptake, Bromodomain-containing protein 4, Apoptosis, Cytarabine

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Ikarugamycin, Darkness-induced degradation of charasomes, Methyl- β -cyclodextrin, Filipin, Lipid rafts

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Activation kinetics, Delayed-rectifier K^+ current, Dexamethasone

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α -Cyclodextrin, Hydrogen bonds, β -CD

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Controllable biocatalyst, N-Isopropylacrylamide, tert-Butyl methacrylate, ϵ -Caprolactone, β -CD, Azobenzene, Microspheres, Food industry

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Natural compounds, Inclusion complex, Functional foods, Bioavailability, Flavonoids, Phenolic derivatives, Coumestans, Triterpenes, Curcumin, Artemisins, Dihydromyricetin

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β -cyclodextrin, Biofilm formation, Bioluminescence, Antibiotic production, 3-oxo-C8-HSL, 3-oxo-C10-HSL

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Triethoxyvinylsilane, N-Isopropylacrylamide, Adsorbing ibuprofen

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Polyurethane surface, Heparin-like moiety, sulfonated β -cyclodextrin, Anticoagulant activity

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Multifunctional microspheres, Thermosensitive, Host-guest interaction, Fe_3O_4 nanoparticle core, Acidic-resistant SiO_2 middle shell, Thermosensitive microgel functional shell, β -CD, Regeneration

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Enantioseparation, Molecularly imprinted hollow fiber membrane, Cross-flow biphasic recognition extraction, D-tartaric acid, Sulfobutyl ether- β -cyclodextrin

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Microencapsulation, Textile testing, Lavender essential oil, Chitosan citrate, β -Cyclodextrin-grafted chitosan

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Water treatment, Tetraethyl orthosilicate, HP- β -CD, β -CD, Pyrene, Anthracene, Phenanthrene, Fluorene, Fluoranthene

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Hydroxypropyl- β -cyclodextrin, Liquid-liquid extraction, Formation constant, Separation factor α

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Surface imprinting, Magnetic property, Mono-6A-deoxy-6-(1-vinylimidazolium)- β -cyclodextrin tosylate, Multiple binding sites

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Anaerobic digestion, Solubilizer, β -Cyclodextrin

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Biotic and abiotic samples, γ -Cyclodextrin, Sodium taurocholate

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Fluorescence intensity, Pharmaceutical formulations, Environmental samples

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Multimodal bioprobes, Alpha-cyclodextrin

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Dipicolylamine, Adenine moiety of ATP, Azobenzene unit, Multipoint recognition system

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Thiolated β -cyclodextrin, Colorimetric sensor

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α -CD, β -CD, γ -CD, Biomolecules, Dopamine, Ascorbic acid, Thioridazine

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Chiral separation, Flavanones, 3,5-Dimethylphenylcarbamoylated β -CD, Amino-functionalized spherical ordered mesoporous silica

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α -Cyclodextrin, Energy transfer

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Synergistic effect, Electrochemical sensor, Co-electrodeposition procedure, Oxidation peak current

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p-Nitrophenol, Replacement of guest molecule

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Per-6-quaternary ammonium- β -cyclodextrin, Molecular adaptor of protein nanopore

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Optimization, Response surface methodology, Organic cosolvents, Heptakis(2,3,6-tri-O-methyl)- β -cyclodextrin

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