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Environmental Application of CD-Modified Titanium Dioxide

TiO_2 occurs in nature as well-known minerals rutile, anatase and brookite. It is produced by mining and purification to obtain pure pigment. It is inexpensive and has no absorption in the visible region. The latter is the cause of the white color. The most important application areas are paints and varnishes as well as paper and plastics, which account for about 80% of the world's TiO_2 consumption. Other pigment applications such as printing inks, fibers, rubber, cosmetic products and foodstuffs account for another 8%. The rest is used in other applications, for instance the production of technical pure titanium, glass and glass ceramics, electrical ceramics, catalysts, electric conductors and chemical intermediates [1]. Its environmental utilization as photocatalyst for degradation of soil and water pollutants is in the focus of research of several groups, but only a few demonstrations of technologies can be found in the literature.

Although TiO_2 has been used as pigment for thousands of years, its safety for humans and for the environment is still extensively studied [2]. When irradiated by ultraviolet light photo-induced redox reactions of adsorbed substances are observed. That is what the environmental applications are based on. Most organic pollutants can be completely decomposed to carbon dioxide and water when TiO_2 is used as the photocatalyst, mainly because of the photogenerated hydroxyl radicals [3].

The photocatalytic reactions are surface reactions and thus the reactant must be adsorbed on the surface of the photocatalyst. On the other hand, the UV light must reach the surface of the catalyst where the reaction takes place. Therefore thin water layers or the surface of the polluted soil can be treated only. Some examples for application [2]: i) the wastewater of disinfection solutions for rice hulls containing highly concentrated agrochemicals was poured on glass wool mats deposited with highly photoactive TiO_2 nanoparticles and spread over a wide area on the ground. The agrochemicals were fully decomposed under sunlight in a few days. The initial total organic carbon (TOC) values of several hundred to thousand ppm decreased to nearly zero in one week. ii) The wastewater of a hydroponic culture system containing also pollutants in addition to nitrogen and phosphorous could be recycled after a UV treatment in a shallow vessel of 10 cm depth covered by porous ceramic plates coated with TiO_2 photocatalyst nanoparticles. The organic pollutants were easily decomposed under solar light, but the nutrient compounds (NO_3^-, PO_4^{3-} and K^+) remained unchanged. iii) volatile organic compounds such as trichloroethylene (TCE) was removed from soil by heating (mixing with calcium oxide) and simultaneous photocatalytic decomposition using a sheet made of paper containing both activated carbon and TiO_2. Fig. 1 shows the schemes of these three technologies redrawn from ref. [2].

Although TiO_2 is used widely, it is a relatively inefficient photocatalyst owing to the recombination of charge-hole pairs generated from the excited TiO_2. Noble metal deposition, ion-doping, dye sensitization, surface reductive treatment and surface chelating are commonly used to improve the properties of TiO_2 [3]. One of the possibilities is to combine it with cyclodextrins.

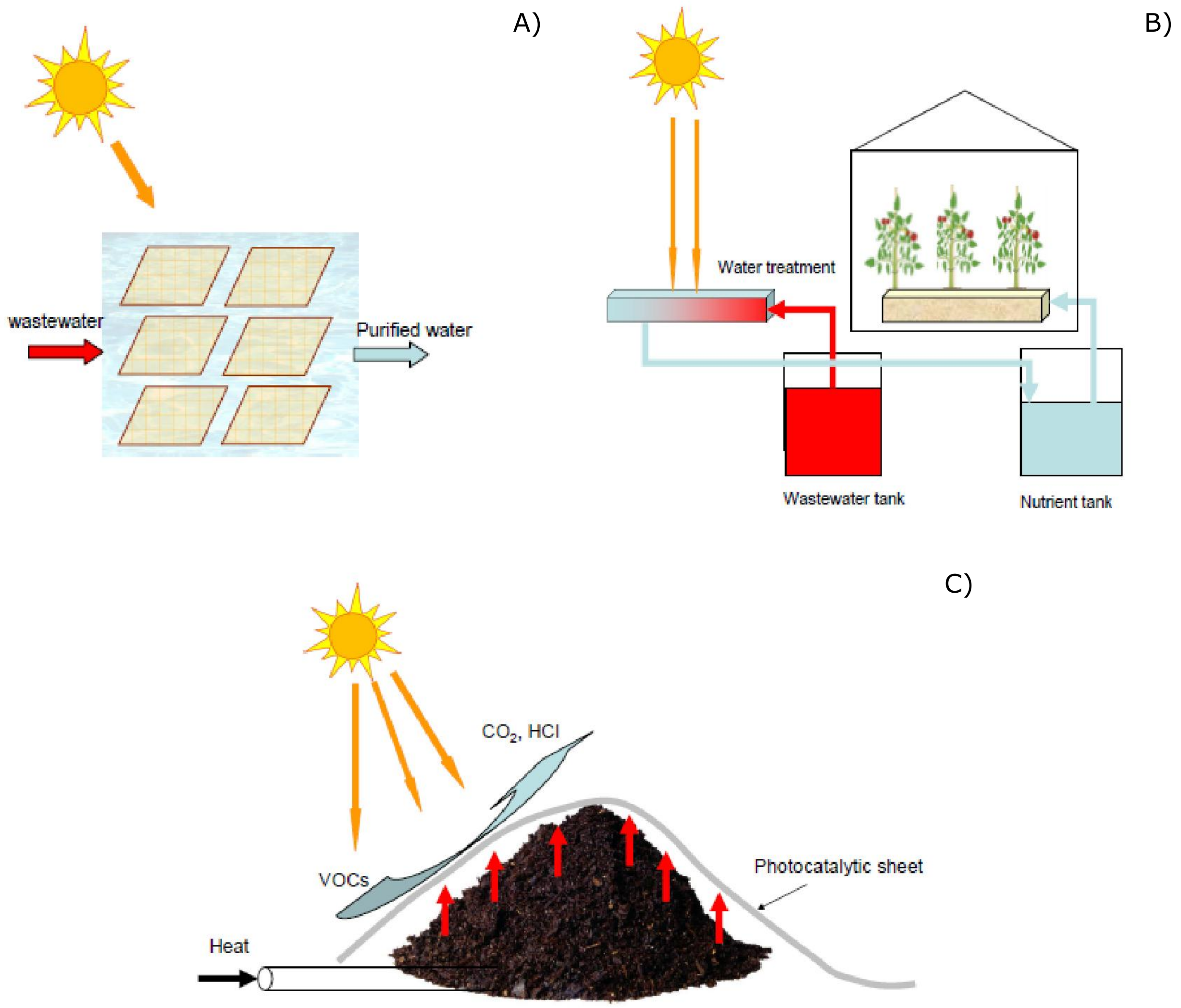


Figure 1.: Water and soil remediation technologies based on TiO_2-catalyzed photodecomposition of pollutants:

A) agricultural wastewater is poured on mats with TiO_2;

B) agricultural wastewater containing nutrients is recycled to the glasshouse after photocatalytic treatment in a vessel with ceramics containing TiO_2;

C) soil contaminated with volatile organic contaminants (VOCs) is covered with a sheet containing TiO_2, as catalyst for the mineralization of VOCs evaporated from the soil by heating (redrawn from ref. [2])

Effect of CDs on the photocatalytic decomposition of pollutants

One of the advantages of the complex formation can be the enhanced solubility of a poorly soluble pollutant. Both in the presence and absence of TiO_2 catalyst, the sensitivity toward decomposition can be changed (increased or decreased) upon complexation depending on the conformation of the guest molecule inside the cavity. If the light-sensitive part of the molecule is located deep inside the cavity it can be protected, if it is protruding out of the cavity the decomposition rate can be enhanced (catalytic effect). The editorial of CDN pre-symposium issue was dedicated to the German scientists and cyclodextrin technologists.

The effect of TiO_2 catalyst can be further improved by the presence of CDs because they adsorb on the surface of TiO_2 and the pollutant included into the cavity gets in closer proximity to the surface of the TiO_2 particles. The adsorption of βCD on the TiO_2 particles from aqueous solutions of increasing βCD concentration follows a saturation curve suggesting a monolayer adsorption process (Fig. 2) [4].

Zhang et al. found that βCD is adsorbed on the TiO_2 particles by its secondary side (Fig. 3). The complexing ability and also the conformation of the inclusion complexes might be different in the case of the adsorbed βCD molecules and those dissolved in the aqueous solution.

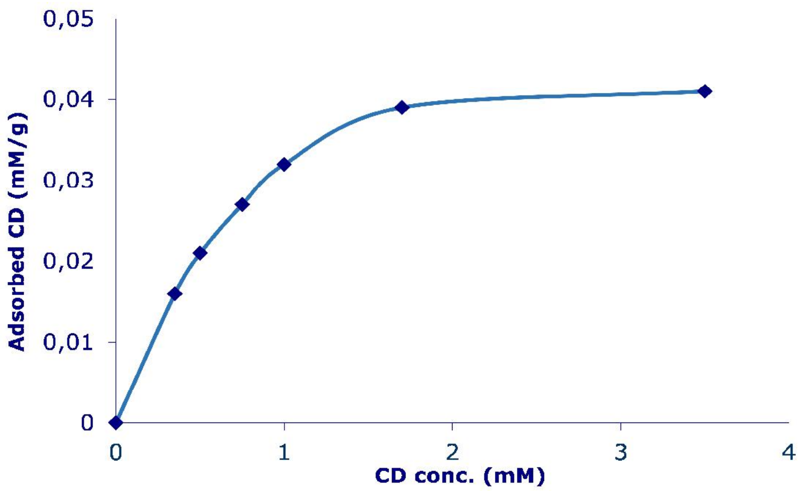


Figure 2.: Adsorption isotherm of βCD on TiO_2 nanoparticles at room temperature [2]

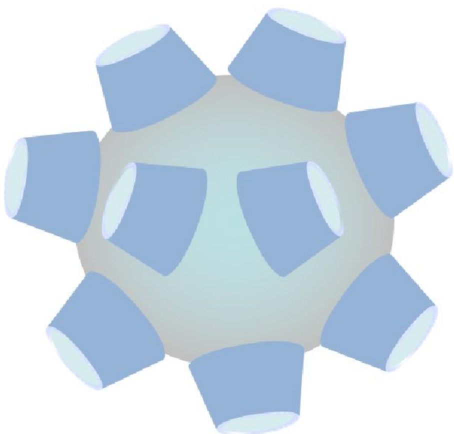


Figure 3.: Scheme of the TiO_2 particles with adsorbed βCD

Improving the colloid stability of TiO_2 nanoparticles

Titanium dioxide colloid system was prepared by slow addition of $TiCl_4$ to βCD solution at 0 °C [5]. The size of the resulting TiO_2 particles determined by TEM was 8 nm and was stabile for several months at pH 1.8 (0.2% TiO_2, 1% βCD). In the absence of βCD TiO_2 precipitated immediately from the dispersion medium. The stabilized colloid provided a photocatalyst-receptor configuration that allows control of the electron transfer process at the interface of the TiO_2 particles. For example, the photoreduction of N,N-dioctyl-4,4'-dipyridinium was significantly more effective in the presence of the βCD-stabilized TiO_2 colloids. The role of complex formation was proved by the decreased rate of photoreduction in the presence of increasing concentration of phenol as competitive guest. Thus βCD has two functions: by adsorption on the surface of the nanoparticles it hinders the aggregation of the nanoparticles (stabilizing function) and provides close proximity for the ligands and the catalyst facilitating the interfacial electron transfer processes (receptor function). βCD plays electron-donating and hole-capturing roles when linked to TiO_2 colloids, that leads to charge-hole recombination restriction and photocatalytic efficiency enhancement.

The effects depend on the guest (pollutant) molecules [6]. The presence of 1-adamantanol in the cyclodextrin cavity does not affect charge separation and trapping because of its unfavorable oxidation potential. However, when ferrocenemethanol was used as the guest molecule the formation of ferrocenium cation was observed, revealing electron transfer from guest molecules to TiO_2 nanoparticles. This results in dissociation of the host-guest assembly because of repulsion of the charged ion from the hydrophobic cavity of βCD into the bulk of the aqueous solution and consequently leads to efficient charge separation and redox chemistry.

The βCD-stabilized TiO_2 colloid system can be successfully sensitized by dyes, which are complexed by the βCD units [7]. For instance, by complexing proflavin dye the photosensitization efficiency for charge injection from the dye to platinium-charged TiO_2 colloids in aqueous media was improved. The uptake of the dye monomer in the βCD cavity increased the surface concentration of proflavin, promoted electron transfer from photoexcited dye molecules to the semiconductor TiO_2, and subsequently, advanced the catalytic H_2 evolution. The quantum yield for H formation in the Pt-TiO_2-βCD system was 3 times larger than that in the Pt-TiO_2 system without βCD.

Similarly to Pt also palladium can be immobilized on TiO_2 colloids resulting in active photocatalysts for the selective reduction of CO_2/HCO_3^- to formate. The active photocatalysts prepared by adsorption of aqueous Pd/βCD colloids onto TiO_2 nanoparticles enhanced the rate of conversion 6 times compared to the Pd-free TiO_2 suspensions [8].

Another composite is the Ag/TiO_2/βCD nanoparticles of 95 nm average particle size, which is stable for more than 15 days and able to catalyze the decomposition of methylene blue dye [9]. Interestingly the βCD-modified nanoparticles improved the rate of decomposition only in a small extent compared to Ag/TiO_2 nanocomposites, and the further increase in the βCD content in the nanoparticles was detrimental for the catalytic efficiency.

Chemical immobilization of βCD on TiO_2

Synthesizing βCD modified TiO_2 colloids is complicated and time-consuming, and the colloids are only stable in acidic conditions. Therefore, it is worthwhile to synthesize a βCD grafted TiO_2 hybrid powder. TiO_2/βCD hybrid nanoparticles were produced by a photoinduced self assembly process [10]. The nanoparticles showed high reactivity for dye pollutant degradation under visible light and simulated solar irradiation.

A Japanese patent describes a general method for immobilization of CDs on metal oxides. First the metal oxide has to be treated to introduce linking groups such as diisocyanates, alkyloxirane halides, dicarboxylic acid halides. Mixing TiO_2 with hexamethylene-diisocyanate in dry dimethylsulfoxide in the first step and adding a dimethylsulfoxide solution of βCD in the second step gave an immobilized βCD [11].

Carboxymethyl-βCD was directly bound to TiO_2 via coupling to butyl titanate then hydrolyzing, ageing and calcining at enhanced temperature. This catalyst has higher efficiency compared with the unmodified TiO_2 in degradation of methyl orange (99.2% and 47.6% after 30 min using the modified and plain TiO_2, respectively) [12].

In order to build chemical bonds between the CD polymer chain and inorganic TiO_2, carboxyl functional group was introduced into copolymer obtained from allyl-βCD and acrylic acid (AA). The photoactivity of Poly(CD-co-AA)/TiO_2 was 2.6 times higher than that of pure TiO_2 colloid in the degradation of methyl orange [13].

Recently combining the unique temperature responsive characters of polymeric material based on N-isopropylacrylamide (NIPAM), a capacity for organic molecular inclusion of maleic anhydride (MAH) modified βCD, and the enhancements in photocatalytic activity of TiO_2 doped with multi-walled carbon nanotubes (MWCNTs), the novel thermosensitive poly(NIPAM-co-MAH-βCD)/(TiO_2-MWCNTs) composite photocatalysts were prepared [14]. The new photocatalyst was successfully applied for the degradation of 2-sec-butyl-4,6-dinitrophenol, model pollutant in aqueous solution under visible light irradiation.

Application for wastewater treatment

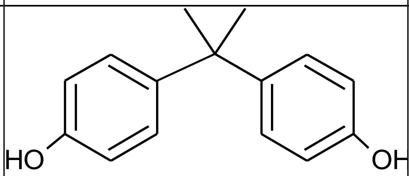
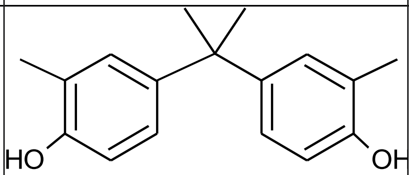
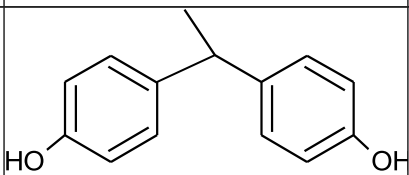
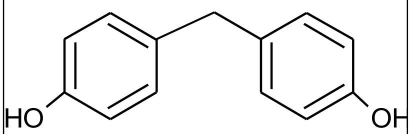
The addition of βCD increased the decolorization (photocatalytic oxidation) rates of a dye (Acid Red 14) by 3-7 times and also of the photocatalytic reduction rates of Cr(VI) by 12-17 times in the solutions containing 0.8 g/L TiO_2 [15, 16]. The effect is explained by enhanced adsorption of the pollutants onto the photocatalyst surface and moderate inclusion-depth in the βCD cavity ensuring accessibility toward the UV light.

The decomposition rate of Rhodamine B was enhanced by 4.6 times with TiO_2/βCD catalyst compared to the unmodified TiO_2 [17]. Similar results were reported for the degradation of Nile red [18].

The photocatalytic degradation of bisphenol A (BPA), C, E and F, endocrine disrupting materials used also for plastic industry was faster in the system containing βCD and TiO_2 compared to that in the system containing only TiO_2 (Table 1). The formation of CO_2 as a result of mineralization was observed during the photodegradation processes.

The enhanced photodegradation of residual pharmaceuticals modeled by paracetamol solutions was also shown by using βCD/TiO_2 hybrid catalyst compared to the pure TiO_2 [19].

Table 1. Mineralization efficiency (%) after 120 min of irradiation in the presence and absence of βCD

Pollutant		**TiO_2 alone**	**TiO_2/βCD**	**Ref**
Bisphenol A		36.7	100	[20]
Bisphenol C		40.6	94.8	[21]
Bisphenol E		23	61	[22]
Bisphenol F		18	48	[23]

Application in soil remediation

The application of CD-modified TiO_2 for soil remediation has not been reported so far. In the following two examples it was studied how the presence of a CD or CD derivative in the soil flushing solution can influence the photodecomposition of the pollutants using non-modified TiO_2 catalyst.

The photocatalytic degradation of pentachlorophenol (PCP) in the flushing solution extracted from contaminated soil was reported by Hanna et al. [24]. While the removal of PCP from the soil was significantly enhanced by using aqueous βCD solution instead of water, the rate of photodecomposition in the presence of TiO_2 catalyst was reduced owing to the protecting effect of the complexation (Fig. 4). Using such combined technologies for soil remediation the optimum CD concentration high enough for improving the extraction and low enough for not remarkably reducing the rate of photodegradation should be found. It should be noted that βCD is also slowly decomposed in this photocatalytic system ($t_{1/2}$: 150 min).

Similar results were obtained by another research group for soils contaminated with polychlorinated biphenyls (PCBs): HPBCD enhanced the extraction of PCBs from the contaminated soil but the photocatalytic decomposition was slowed down [25, 26]. The overall efficiency of the combined technology (soil flushing + photocatalytic degradation), however, was still enhanced by HPBCD.

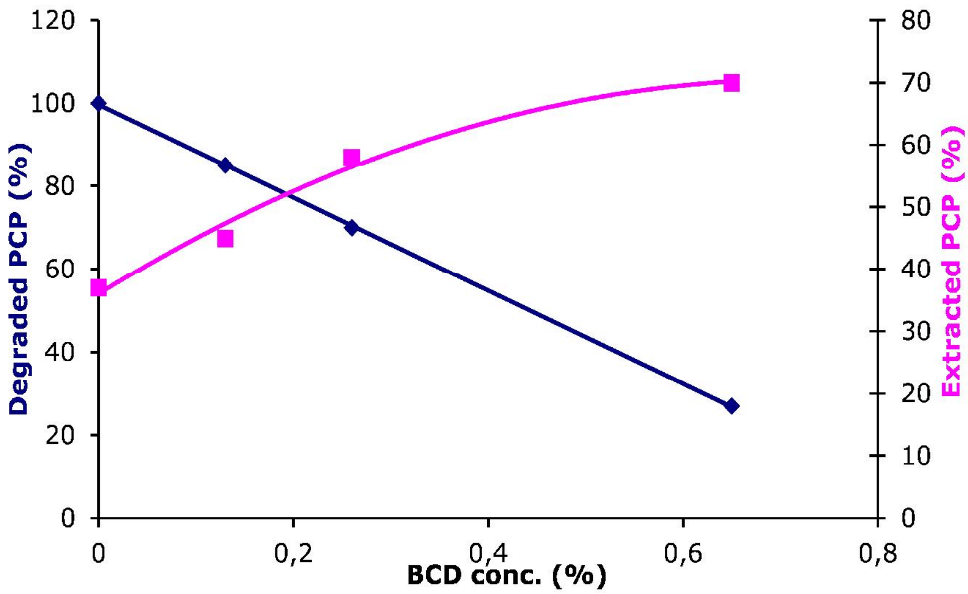


Figure 4.: Effect of βCD concentration on the extraction of PCP from contaminated soil using aqueous βCD flushing solution and subsequent photocatalytic degradation of PCP in this solution [drawn from the data in ref. 24]

Conclusions

1. The colloid stability of TiO_2 nanoparticles can be improved by βCD.

2. Various hybrid CD/TiO_2 systems were prepared and found to be effective photocatalysts with improved properties.

3. Several applications of CD-modified TiO_2 for remediation of various model wastewaters have been reported.

4. The presence of CD in the soil flushing solution improves the extraction, but may decelerate the photodecomposition with TiO_2 catalyst.

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Ex vivo penetration, (2-Hydroxy)propyl-β-cyclodextrin, Antifungal activity, Itraconazole

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Chitosan, Hydrocortisone, Drug Delivery

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Chitosan, Films Thickness, Swelling Index, Mechanical Properties, Ocular

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Iontophoresis, Permeation Enhancer, Propofol, Sulfobutyl Ether-β-cyclodextrin

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Cerebellum, Granule Neuron Proliferation, (2-Hydroxy)propyl-β-cyclodextrin, Niemann Pick C1, Developmental Defect

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3-(Diethylamino)propyl, pH-Responsive, Poly(γ-cyclodextrin), Doxorubicin

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Glyceryl Monooleate, Phytantriol, β-Cyclodextrin

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Nanoparticles, Time-resolved Fluorescence, van der Waals Interaction

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Antimicrobial activity, Essential Oil, Microencapsulation, Minced Pork

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Doxorubicin, Host-guest Interactions, Prodrug Micelles, β-CD-hydrazone-DOX, PEG-Fc, Fluorescence Microscopy, Flow Cytometry Analysis

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Frozen Stability, Meropenem, Vitrification, (2-Hydroxy)propyl-β-cyclodextrin, Glass Transition Temperature

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Review, Encapsulation Efficiency, Pore Control Motifs, Release Rate

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Bioavailability, Dissolution Rate, Nanoparticles, Flavonol

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Diffusion Coefficient, Aquaporin, K-Space Image Correlation Spectroscopy, MDCK, Micropatterning

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Adhesion Molecules, Carbon Monoxide, Endothelial Cells, Enzyme-triggered CORMs

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Anticancer, NMR Spectroscopy, DSC, TG, Photometry

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Anthrax Toxin Receptor 2, Cholesterol, ERK Phosphorylation, MMP-2, MT1-MMP

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AITC, Antibacterial Activity, Electrospinning, Nanofibers, Release, Sustained Release, PVA Nanofiber

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GC, Sensory Evaluation, Maltodextrin, Volatiles, (2-Hydroxy)propyl-β-cyclodextrin, Emulsion, Secondary Lipid Oxidation Product

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β-Cyclodextrin, Carvacrol, Microfibrillated Cellulose, Release Study, Sustained Release Packaging

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Aroma, Capillary Electrophoresis, Catechins, Chiral Analysis

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Fouling, Hydrophilicity, Mixed Matrix Membranes, Natural Organic Matter, Rejection

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Ageing Effect, Benzo[a]pyrene, Extractability, Oral Bioavailability, Soil Properties, Swine Model

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Review, Delivery Systems, Encapsulation, Food Bioactives, Nanocarriers, Guar Gum

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Adsorption Kinetics of Cu^{2+}, Mechanism, Pseudo-second-order Rate Equation

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Lack of Surfactant, Flocculation, Electrostatic Interaction, Mechanism

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Ibuprofen, Micro-pollutants, Nanofiltration, Sorption, β-Cyclodextrin, Total Organic Carbon, Regeneration of Nanofilter

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Green Synthesis, Microwave Irradiation, β-Cyclodextrin Polymers, Hexamethylene Diisocyanate

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Bioassay, Biological Post-treatment, Electro-Fenton, HPCD, Hydroxyl radical, Polycyclic Aromatic Hydrocarbons, Electrochemical Treatment

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Alkylphenols, Emerging Pollutants, Treatment Methods, Adsorption-oriented Process

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Adsorption, Arsenic, Groundwater., Hydrous Ferric Oxide, β-cyclodextrin

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Activated Carbon, in-situ Soil Flushing, Mineral Oil, Soil Aquifer Remediation

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Bioelectrolysis, Biohydrogen, Pretreatment, Volatile Fatty Acid, Waste Activated Sludge, Hydrogen Production Rate

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CD/MMT/Iron Oxide Composite, Magnetic Separation, Sorption, Thermodynamic Data, Uranyl(VI)

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Mixed-contaminated Site, Soil Washing, Tenax Extraction, Soil Microorganism, Residual Pollutant, Combined Cleanup Strategy

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Carboxymethyl Chitosan, Carboxymethyl-β-cyclodextrin, Mixed-Contaminated Sites, Soil Washing, Tenax Extraction

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X70 Steel, Electrochemical Impedance Spectroscopy, Polarization, SEM, Acid Inhibition

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Adsorption, Corrosion, Polymer, SEM, Q235 Carbon Steel, Protection Film

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Stationary Phase, Electrochromatography

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Amantadine, Fluorescence Recovery, Graphene Oxide, Rhodamine6G, β-Cyclodextrin

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Host-guest Interaction, Multicolor, Self-assembly, Adamantane-labeled Fluorescein and Rhodamine B, β-Cyclodextrin Polymer

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Chiral Selector, Chiral Stationary Phase, GC, HPLC, Inclusion Complex, Mechanism

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Capillary Electrophoresis, Chiral Ionic Liquid, Chiral Separation, First-order Derivative Electropherogram, Quinine/Quinidine, Cinchonine/Cinchonidine, (2-Hydroxy)propyl-β-cyclodextrin

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Inclusion Interaction, Solid Phase Extraction, β-cyclodextrin Crosslinked Polymer

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Tel:(361) 347-6060
Fax: (361) 347-6068
e-mail: cyclolab@cyclolab.hu